

**Evaluation of Herbicides as Desiccants for Lentil (*Lens culinaris* Medik)  
Production**

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By  
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## Abstract

The indeterminate nature of lentil (*Lens culinaris* Medik), in conjunction with adverse field conditions, can lead to varying degrees of maturity among plants at harvest. This variable maturity may have a negative influence on lentil production and can delay harvest. Desiccants are currently used to improve lentil crop dry-down. However, applying desiccants too early may result in reduced crop yield and quality, and also leave unacceptable herbicide residues in lentil seeds. In addition, only four herbicides (glyphosate, diquat, saflufenacil, and glufosinate) are registered as desiccants for lentil desiccation in Canada, which limits options for growers. Therefore, the objectives of this thesis were i) to determine the importance of desiccant application timing in affecting crop yield and quality, as well as herbicide residues and ii) to determine whether additional desiccants applied alone or tank-mixed with glyphosate provide better crop desiccation. Field trials were conducted at Saskatoon and Scott, Saskatchewan, from 2012 to 2014. In the application timing trial, glyphosate or saflufenacil alone, or glyphosate+saflufenacil generally decreased seed yield, thousand seed weight, and crop dry-down, and increased herbicide residue levels at earlier application timings. For example, when applied at 60% seed moisture, saflufenacil reduced yield and thousand seed weight by 22% and 10%, respectively, and resulted in glyphosate and saflufenacil residues greater than 2.0 and 0.03 ppm, respectively. Although there were no reductions in yield and thousand seed weight when desiccants were applied at 50% or 40% seed moisture, glyphosate residue exceeded 2.0 ppm. Application of desiccants at 20 or 30% seed moisture content had no effect on yield, thousand seed weight, or herbicide residues. These results indicate that desiccant application timing is critical, and should not be made before 30% seed moisture. In a second study, glufosinate and diquat tank mixed with glyphosate were the most consistent desiccants and provided optimal crop dry-down without reducing yield and thousand seed weight, and effectively reduced glyphosate residue. The other herbicides tested (pyraflufen-ethyl and flumioxazin) were found to be poor options for growers as they had sub-optimal crop desiccation and did not affect glyphosate residue.

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## Table of Contents

Permission to Use .....	i
Abstract.....	ii
Acknowledgements .....	iii
List of Tables .....	vi
List of Figures.....	viii
List of Abbreviations .....	x
<b>1.0 Introduction.....</b>	<b>1</b>
<b>2.0 Literature Review .....</b>	<b>4</b>
2.1 Lentil growth habit, global production, and uses .....	4
2.2 Lentil in Canada .....	5
2.3 Lentil harvesting .....	7
2.4 Weed competition in lentil .....	8
2.5 Pre-harvest desiccants.....	9
2.6 Properties of chemical harvest aids.....	11
2.7 Limitations of chemical harvest aids.....	14
<b>3.0 Impact of Glyphosate and Saflufenacil Application Timing on Lentil Seed Yield, Quality and Herbicide Residues .....</b>	<b>17</b>
3.1 Introduction .....	17
3.2 Hypotheses.....	19
3.3 Materials and Methods .....	19
3.3.1 Experiment site and design .....	19
3.3.2 Experimental procedure .....	20
3.3.3 Data collection.....	24
3.3.4 Statistical analysis .....	26
3.4 Results and Discussion .....	26
3.4.1 Lentil desiccation .....	26
3.4.2 Seed yield .....	30
3.4.3 Thousand seed weight .....	32
3.4.4 Harvest straw moisture content .....	34
3.4.5 Herbicide residues .....	36
3.5 Conclusions.....	39
<b>4.0 Evaluation of Various Herbicides as Potential Desiccants in Lentil .....</b>	<b>41</b>
4.1 Introduction .....	41
4.2 Hypotheses.....	43
4.3 Materials and Methods .....	43
4.3.1 Experimental site and design.....	43
4.3.2 Experimental procedure .....	43
4.3.3 Data collection.....	46
4.3.4 Statistical analysis .....	47
4.4 Results and Discussion .....	47
4.4.1 Lentil desiccation .....	47
4.4.2 Seed yield and thousand seed weight .....	51
4.4.3 Harvest straw moisture content .....	53
4.4.4 Glyphosate residue .....	55

4.5 Conclusions.....	58
5.0 General Discussion.....	60
5.1 The use of desiccants in lentil .....	60
5.2 Management implications .....	62
5.3 Future research.....	63
6.0 Literature Cited .....	65

## List of Tables

<b>Table 2.1</b> Maximum Residue Limits (MRLs) in parts per million (ppm) in lentil for European Union, International CODEX (International standards), Japan, United States and Canada. Adapted from Global MRL Database (Bryant Christie Inc., 2015).....	15
<b>Table 3.1</b> Herbicide treatments, rates, and application timings (% seed moisture content) for each herbicide treatment evaluated at Saskatoon and Scott, SK from 2012 to 2014.....	22
<b>Table 3.2</b> Dates of application timings and environmental conditions (temperature, relative humidity and wind) for each herbicide treatment in timing trials at Saskatoon and Scott, SK from 2012 to 2014.....	23
<b>Table 3.3</b> P-values derived from analysis of variance for area under desiccation progress curve (AUDPC), lentil seed yield, thousand seed weight (TSW), straw moisture, glyphosate residue (GR), and saflufenacil residue (SR) as influenced by herbicide (H) and application timing (T) at Saskatoon and Scott, SK from 2012 to 2014.....	27
<b>Table 3.4</b> P-values derived from analysis of variance illustrating fixed effects (herbicide and application timing) for the area under desiccation progress curve (AUDPC) at Saskatoon and Scott, SK from 2012 to 2014.....	27
<b>Table 3.5</b> Contrast statements of area under desiccation progress curve (AUDPC) represent comparisons for each herbicide treatment at various application timings (% seed moisture content), showing the estimate of difference between means at Saskatoon and Scott, SK from 2012 to 2014.....	30
<b>Table 3.6</b> Contrast statements of yield, thousand seed weight (TSW), and straw moisture, represent comparisons for each herbicide treatment at various application timings (% seed moisture content), showing the estimate of difference between means at Saskatoon and Scott, SK from 2012 to 2014.....	31

<b>Table 3.7</b> Contrast statements of glyphosate residue (GR) and saflufenacil residue (SR), represent comparisons for each herbicide treatment at various application timings (% seed moisture content), showing the estimate of difference between means at Saskatoon and Scott, SK from 2012 to 2014.....	38
<b>Table 4.1</b> Herbicide treatments and rates for each herbicide treatment evaluated at Saskatoon and Scott, SK from 2012 to 2014.....	45
<b>Table 4.2</b> Dates of application timings and environmental conditions (temperature, relative humidity and wind) for each treatment at Saskatoon and Scott, SK from 2012 to 2014.....	46
<b>Table 4.3</b> P-values derived from analysis of variance demonstrating area under desiccation progress curve (AUDPC), seed yield, thousand seed weight (TSW), straw moisture, and glyphosate residue (GR), as influenced by herbicide treatments at Saskatoon and Scott, SK from 2012 to 2014.....	48
<b>Table 4.4</b> Tukey's HSD means comparison of lentil areas under desiccation progress curve (AUDPC) at Saskatoon and Scott, SK from 2012 to 2014. Estimate statements represent differences between herbicide treatments in lentil desiccation.....	49
<b>Table 4.5</b> Tukey's HSD means comparison of seed yield and thousand seed weight (TSW) at Saskatoon and Scott, SK from 2012 to 2014. Estimate statements represent differences between herbicide treatments in seed yield and TSW.....	52
<b>Table 4.6</b> Tukey's HSD means comparison of harvest straw moisture at Saskatoon and Scott, SK from 2012 to 2014. Estimate statements represent differences between herbicide treatments in harvest straw moisture.....	54
<b>Table 4.7</b> Tukey's HSD means comparison of glyphosate residue (GR) at Saskatoon and Scott, SK, in 2012 and 2013. Estimate statements represent differences between herbicide treatments in glyphosate residue.....	57



## List of Figures

**Figure 2.1** Lentil production data in Canada from 2005 to 2014.....6

**Figure 3.1** Relationship between area under desiccation progress curve (AUDPC) and application timing (% seed moisture content) at (A) Saskatoon 2012, (B) Saskatoon 2013, (C) Saskatoon 2014, (D) Scott 2013, and (E) Scott 2014. Regression equation across three herbicide treatments at (B) Saskatoon 2013:  $Y = -6.06x + 1327.70$ ,  $R^2 = 0.967$ ,  $P = 0.0167$ . No relationship was observed for each herbicide at any other site-year. Error bars represent one standard error of the mean.....29

**Figure 3.2** Relationship between seed yield and application timing (% seed moisture content) across five site-years in Saskatchewan. Regression equation for the tank mixture of glyphosate+saflufenacil:  $Y = -0.2x^2 + 3940.4$ ,  $R^2 = 0.7935$ ,  $P = 0.0426$ . No relationship was observed for glyphosate or saflufenacil applied alone. Points ( $\blacktriangle$ ) represent glyphosate; points ( $\diamond$ ) represent saflufenacil; points ( $\bullet$ ) represent glyphosate+saflufenacil. Control yield was  $3358.0 \pm 252.0 \text{ kg ha}^{-1}$ . Error bars represent one standard error of the mean.....31

**Figure 3.3** Relationship between thousand seed weight and application timing (% seed moisture content) across five site-years in Saskatchewan. Regression equation for saflufenacil:  $Y = -0.0015x^2 + 0.0469x + 39.3940$ ,  $R^2 = 0.9680$ ,  $P = 0.032$ ; regression equation for the tank mixture of glyphosate+saflufenacil:  $Y = -0.0004x^2 + 39.4278$ ,  $R^2 = 0.7969$ ,  $P = 0.0415$ . No relationship was observed between TSW and glyphosate applied alone. Points ( $\blacktriangle$ ) represent glyphosate; points ( $\diamond$ ) represent saflufenacil; points ( $\bullet$ ) represent glyphosate+saflufenacil. Control TSW was  $40.6 \pm 0.8 \text{ g}$ . Error bars represent one standard error of the mean.....33

**Figure 3.4** Relationship between harvested straw moisture content and application timing (% seed moisture content) across five site-years in Saskatchewan. Regression equation for glyphosate:  $Y=0.0046x^2+21.6778$ ,  $R^2= 0.7782$ ,  $P=0.0477$ ; regression equation for saflufenacil:  $Y=0.0050x^2+24.3442$ ,  $R^2= 0.8185$ ,  $P=0.0348$ . No significant relationship was observed between the tank mixture of glyphosate+saflufenacil and straw moisture. Points (▲) represent glyphosate; points (◇) represent saflufenacil; points (●) represent glyphosate+saflufenacil. Control straw moisture was  $34.8 \pm 4.1\%$ . Error bars represent one standard error of the mean.....35

**Figure 3.5** Relationship between herbicide residue and application timing (% seed moisture content) across three site-years in Saskatchewan. (A) Regression equation of glyphosate residue across two herbicides:  $Y=0.0019x^2-0.2377$ ,  $R^2= 0.9339$ ,  $P= 0.0074$ . (B) Regression equation of saflufenacil residue across two herbicides:  $Y=0.0000072x^2+0.0001433$ ,  $R^2= 0.9372$ ,  $P= 0.0068$ . Error bars represent one standard error of the mean.....37

## **List of Abbreviations**

AAFC = Agriculture and Agri-food Canada  
AMPA = Aminomethylphosphonic Acid  
ANOVA = Analysis of variance  
AUDPC = Area under desiccation progress curve  
CDC = Crop Development Center  
DAA = Days after application  
EPSP = 5-enolpyruvylshikimate-3-phosphate  
GR = Glyphosate residue  
H = Herbicide  
HSD = Honest significant difference  
HPLC = High performance liquid chromatography  
LC-MS = Liquid chromatography-mass spectrometry  
MRL = Maximum residue limit  
ppm = Parts per million  
PPO = Protoporphyrinogen IX oxidase  
SK = Saskatchewan  
SR = Saflufenacil residue  
SY = Site-year  
T = Application timing  
TSW = Thousand seed weight

## 1.0 Introduction

Lentil (*Lens culinaris* Medik) is a member of the legume family (Muehlbauer et al., 1985; Muehlbauer and McPhee, 2005; Sandhu and Singh, 2007; Erskine et al., 2009; Boye, 2013). It has long been considered part of a healthy diet due to its high protein, carbohydrate, energy, and vitamin content (Muehlbauer et al., 1985; Grusak, 2009; Boye, 2013). Canada has been the leading lentil exporter in the world market, marketing lentil to more than 100 countries (Agricultural and Agri-Food Canada, 2013a; Saskatchewan Ministry of Agriculture, 2010; Pulse Canada, 2014). Saskatchewan is the major lentil-growing province in Canada because of its cool temperature and fertile soils for lentil growth (Saskatchewan Ministry of Agriculture, 2010; Statistics Canada, 2014).

Lentil plants have an indeterminate growth habit, resulting in variable maturity at harvest (Saxena, 2009), which may result in a delayed harvest (Saskatchewan Pulse Growers, 2011; Alberta Pulse Growers, 2013). Late-season weeds and unfavorable weather are the other factors that can delay harvest, often reducing lentil seed yield and quality (Yenish et al., 2009; Saskatchewan Pulse Growers, 2011; Alberta Pulse Growers, 2013). In order to facilitate lentil dry-down and prevent weed interference, desiccants are widely used by producers at late crop growth stages (Riethmuller et al., 2005; Ali et al., 2009; Saskatchewan Pulse Growers, 2011; Schemenauer, 2011; Alberta Pulse Growers, 2013). Currently, glyphosate, diquat, saflufenacil and glufosinate are registered for lentil desiccation (Saskatchewan Ministry of Agriculture, 2014; Fleury, 2015). Glyphosate is a popular pre-harvest herbicide in western Canada due to excellent perennial weed control (Baylis, 2000; Schemenauer, 2011; Saskatchewan Pulse Growers, 2011; Saskatchewan Ministry of Agriculture, 2014). It must be translocated to target sites via the phloem to cause plant mortality (Devine et al., 1993). Thus, it is considered a slow-acting herbicide, resulting in slow crop desiccation (Baylis, 2000; Duke and Powles, 2008; Saskatchewan Ministry of Agriculture, 2014). In contrast, diquat, glufosinate and saflufenacil are contact or contact-like herbicides (Devine et al., 1993), and affect the crop more rapidly than glyphosate (Schemenauer, 2011). However, they frequently do not provide perennial weed control because they have very limited-to-no translocation in plants (Schemenauer, 2011). Thus, it may be beneficial for producers to apply a tank-mixture of glyphosate and a contact herbicide to achieve rapid, uniform crop desiccation and adequate weed control.

Herbicide residues in the seed can be a major concern for exporters when desiccants are applied at advanced crop growth stages. Importing countries often will reject lentils if residues exceed the maximum residue limit (MRL) (Bryant Christie Inc., 2015). This issue received sizeable attention in 2011 as Canadian lentils were not accepted by the European Union because glyphosate residue was over the established MRL of 0.1 part per million (ppm) (Pratt, 2011). Likewise, Canadian dry beans (*Phaseolus vulgaris* L.) were rejected by Japan because excess levels of glyphosate residue were detected in seeds (Sprague, 2012). Timely application of desiccants is therefore crucial, as improper application timing can result in reductions in seed yield and quality (Whigham and Stroller, 1979; Azlin and McWhorter, 1981; Cerkauskas et al., 1982; Ratnayake and Shaw, 1992; Ellis et al., 1998; Bennett and Shaw, 2000a; Darwent et al., 2000; Wilson and Smith, 2002; Boudreaux and Griffin, 2011; Soltani et al., 2013), and leave unacceptable herbicide residues in seeds (Cessna et al., 1994, 2000; 2002). Desiccants should be applied when crops are close to or at physiological maturity and nearing harvest maturity (Saskatchewan Ministry of Agriculture, 2014).

Currently, few herbicides are available for lentil desiccation in Canada. It is possible that Canadian producers could improve their competitive ability in the global marketplace if additional desiccants were available. Pyraflufen-ethyl and flumioxazin (contact herbicides) have not been registered as desiccants in lentil, but they are used in desiccating other crops. For example, pyraflufen-ethyl effectively desiccated potato without adverse impacts on harvested tuber stem quality (Ivany, 2005; Nichino Europe Co. Limited, 2012). Flumioxazin enhanced dry bean desiccation and provided good residual weed control (Valent Canada, Inc., 2009; Soltani et al., 2013). Therefore, these two herbicides have the potential to be desiccants for lentil production.

Research on desiccants has been conducted in legume plants such as soybean (*Glycine max* L.), dry bean, and field pea (*Pisum sativum* L.) (Retnayake and Shaw, 1992; Ellis et al., 1998; Bennett and Shaw, 2000a; Willson and Smith, 2002; Baig et al., 2003; Soltani et al., 2013; McNaughton et al., 2015). However, there is little research on the effects of desiccants in lentil production, particularly on lentil desiccation, seed yield, and herbicide residues. The effective application timing of desiccants is important knowledge for producers to avoid unacceptable herbicide residues, and to retain optimal crop yield and quality. Thus, the overall objective of this thesis was to improve the use of desiccants in lentil production in western Canada. Specifically,

the thesis objectives were two-fold: 1) to determine the effect of desiccant application timing on lentil desiccation, seed yield, quality, and herbicide residues and 2) to determine the response of lentil to various desiccants applied alone or in tank mixture with glyphosate.

## 2.0 Literature Review

### 2.1 Lentil growth habit, global production, and uses

Lentil (*Lens culinaris* Medik) is an annual plant species with a taproot (Saxena, 2009; Saskatchewan Ministry of Agriculture, 2010; Saskatchewan Pulse Growers, 2011). The general height of lentil ranges from 20 to 30 cm, but it can reach upwards of 75 cm depending on environmental conditions (Muehlbauer et al., 1985). Similar to other pulse crops, it exhibits variation at maturity because of its indeterminate growth habit, which can be significantly influenced by environmental conditions and lentil variety (Saxena, 2009; Government of Saskatchewan, 2010; Saskatchewan Pulse Growers, 2011). As a result, both immature and mature pods on a plant may be observed at the same time (Saxena, 2009; Government of Saskatchewan, 2010; Saskatchewan Pulse Growers, 2011). Generally, varieties with longer maturities produce a more indeterminate growth habitat (Saskatchewan Pulse Growers, 2011). Lentil varieties exhibit variation in seed size, as well as hairiness and colour of the leaves, flowers, and seeds (Sandhu and Singh, 2007; Muehlbauer et al., 2009). Normally, lentil can be divided into two types by size: large seeded (macrosperma) and the small to medium seeded (microsperma) (Sandhu and Singh, 2007; Muehlbauer et al., 2009; Saskatchewan Ministry of Agriculture, 2010). Lentil can also be classified based on seed coat and cotyledon colour, which includes green, red, or brown lentils (Sandhu and Singh, 2007; Muehlbauer et al., 2009; Saskatchewan Ministry of Agriculture, 2010).

Lentil is one of the oldest domesticated plants under cultivation (Muehlbauer et al., 1985; Harlan, 1992), and is believed to have been first planted in southwest Asia (Saskatchewan Ministry of Agriculture, 2010). It is among the major pulse crops grown worldwide (McNeil et al., 2007). World lentil production was about 4.9 million tonnes in 2013 (FAOSTAT, 2015).

Lentil is consumed in many parts of the world as an important part of daily food intake because of its high nutritional level (Singh, 1999; Ghosh et al., 2007; Urbano et al., 2007; Grusak, 2009; Boye, 2013). Its seeds contain substantial protein, minerals, and vitamins, which are important for human health (Bhatty, 1988; Urbano et al., 2007; Grusak, 2009; Boye, 2013). More specifically, lentil has the highest protein level (following soybeans) among vegetables (Bhattacharya et al., 2005). Lentil provides most essential amino acids, some of which are

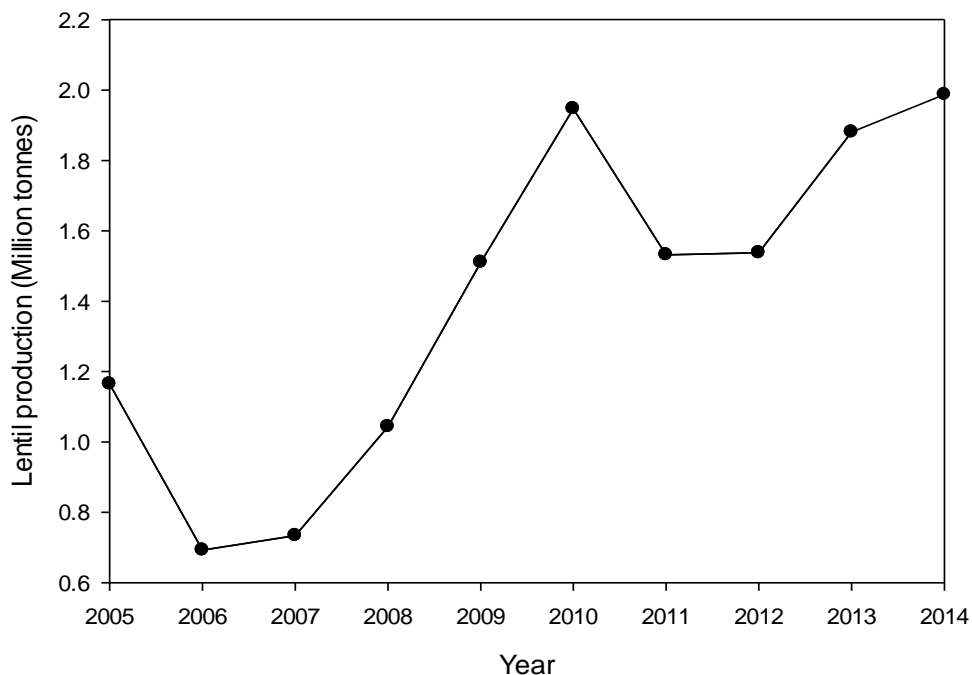
required by humans but are difficult to obtain in cereal-based diets (Erskine et al., 2009). Lentil contains greater levels of essential amino acids such as lysine, arginine and leucine, than other cool season pulse crops (Erskine et al., 2009). Its seed also contains various minerals including potassium, phosphorus, calcium, magnesium, copper, iron and zinc (Bhatty, 1988; Urbano et al., 2007; Grusak, 2009). It is high in water-soluble vitamins, especially B vitamins (Bhatty, 1988; Urbano et al., 2007; Grusak, 2009). Lentil has a relatively short cooking time, especially small lentil varieties, compared with other dried grain legumes (Muehlbauer and McPhee, 2005; Yadav et al., 2007).

Apart from human consumption, lentil straw can also be used to feed livestock (Erskine et al., 1990). Muehlbauer et al. (1985) reported that lentil straw contains approximately 4.4% protein and 50% carbohydrate. Furthermore, there is a symbiotic relationship between lentil and *Rhizobium* bacteria, which can provide lentil with biologically fixed nitrogen (Government of Saskatchewan, 2007; Quinn, 2009). In turn, the *rhizobium* bacteria receive nutrients and water from lentil (Government of Saskatchewan, 2007; Quinn, 2009). Consequently, lentil demands less nitrogen fertilizer than other crops, such as cereals and oilseeds (AAFC, 2013b).

## **2.2 Lentil in Canada**

Canada has been the largest lentil exporting country in the world since 2005 (AAFC, 2013b; Saskatchewan Ministry of Agriculture, 2010; Pulse Canada, 2014). In the past decade, lentil production in Canada has increased from 1.1 to 2.0 million tonnes as growers have increased the number of hectares on which lentil is grown, largely due to increased production efficiency (Figure 2.1) (FAOSTAT, 2015; Statistics Canada, 2015). Most (99%) of the lentils grown in Canada are produced in Saskatchewan, with some grown in southern Alberta and Manitoba (Pulse Canada, 2014). The Brown, Dark Brown, and Black soil zones of Saskatchewan provide adequate soil and climatic conditions for lentil growth, although the majority of lentils are grown in the Brown and Dark Brown soils zones (Saskatchewan Pulse Growers, 2011; Statistics Canada, 2014).





**Figure 2.1** Lentil production data in Canada from 2005 to 2014.

Red lentil is more predominant than the other classes and accounts for about 60% of lentil production in global trade (Erskine et al., 2009; Saskatchewan Ministry of Agriculture, 2010; Vandenberg and SK Crops Branch, Saskatchewan Agriculture, 2010; Statistics Canada, 2014). Although large green lentil is the main seed coat color in western Canada (AAFC, 2013a and 2013b), red lentil is becoming more popular for lentil producers in western Canada due to increased demand from the world market (Government of Saskatchewan, 2010; Vandenberg and SK Crops Branch, Saskatchewan Agriculture, 2010). CDC Maxim, a red lentil cultivar, has gained popularity in Canada during recent years (Government of Saskatchewan, 2010). CDC Maxim has a grey seed coat and small seed size, similar to CDC Redberry. This lentil variety is high yielding compared with other red lentil varieties (Vandenberg and SK Crops Branch, Saskatchewan Agriculture, 2010) and is also resistant to ascochyta blight (*Ascochyta lentis* Vassilievsky) and anthracnose (*Colletotrichum truncatum* (Schwein.) Andrus and Moor) (Government of Saskatchewan, 2010). In addition, CDC Maxim has been bred to tolerate imidazolinone (Group 2) herbicides, which aids in weed control (Government of Saskatchewan, 2010).

Lentil plants are well adapted to a low temperature environment and also tolerate drought better than other legume crops (Andrews and McKenzie, 2007; Saskatchewan Ministry of Agriculture, 2010; Saskatchewan Pulse Growers, 2011). Lentils are sown in early spring in Canada, and generally reach maturity at 75 to 100 days after planting (Saxena, 2009). Lentil can grow well in soil pH ranging from 6.0 to 8.0 (Ali et al., 2009; Saskatchewan Pulse Growers, 2011), but is sensitive to excessive water and high soil salinity (Materne and Siddique, 2009; Saskatchewan Pulse Growers, 2011). Diseases are another factor that can lower seed yield and quality. For example, ascochyta blight (*Ascochyta lentis* Vassilievsky) and anthracnose (*Colletotrichum truncatum* (Schwein.) Andrus and Moor) can cause considerable economic losses (Chen et al., 2009; Saskatchewan Ministry of Agriculture, 2010; Saskatchewan Pulse Growers, 2011).

### **2.3 Lentil harvesting**

Because of its indeterminate nature, harvesting lentil can be a challenge. The extent of that challenge can also vary with cultivar due to differences in maturity. There are two major advantages of early-maturing lentil cultivars compared with later-maturing cultivars (Saskatchewan Ministry of Agriculture, 2010). First, shorter maturation times can prevent flowering at mature stages, which would occur late in the season (Saskatchewan Ministry of Agriculture, 2010). Secondly, early maturing lentil often matures prior to fall frost (Saskatchewan Ministry of Agriculture, 2010).

For most lentil growers, harvesting the crop during the cool fall temperatures can be challenging (Muehlbauer et al., 2002; Saskatchewan Ministry of Agriculture, 2010; Saskatchewan Pulse Growers, 2011). Downgrading can result from delayed harvest under cool environmental conditions, resulting in decreased seed yield and quality (Riethmuller et al., 2005; Saskatchewan Pulse Growers, 2011; Alberta Pulse Growers, 2013). Lentil pods can shatter and drop from ripe pods when harvest is delayed (Riethmuller et al., 2005; Saskatchewan Pulse Growers, 2011; Alberta Pulse Growers, 2013). Moden et al. (1986) reported that one third of lentil yield loss resulted from mature pod shatter. Yield is generally maximized by preventing shatter loss instead of waiting for less mature pods to dry down (Saskatchewan Pulse Growers, 2011). Nevertheless, plants are likely to be influenced by environmental conditions such as those

that delay harvest (Riethmuller et al., 2005). This can be problematic as harvest delays can produce lentil seeds with low moisture content, which are more susceptible to mechanical damage when harvested (Tang et al., 1992). Similar issues exist in soybean production as Philbrook and Oplinger (1989) observed that decrease of soybean yield increased steadily at a rate of 0.2 % per day due to mechanical damage in late-harvested soybeans.

In light of the aforementioned issues, most growers try to harvest lentil as early as possible (Ali et al., 2009; Saskatchewan Pulse Growers, 2011; Alberta Pulse Growers, 2013). However, the uneven maturity in lentil can reduce harvest efficiency (Ghosh et al., 2007; Saskatchewan Pulse Growers, 2011). In addition, lentil can revert back to vegetative growth under wet weather, and it will keep growing vegetatively until adverse conditions terminate growth (Government of Saskatchewan, 2010; Saskatchewan Pulse Growers, 2011). Although producers can wait for lentil crops to dry-down evenly, longer dry-down periods may pose a higher risk of disease infection, lead to pod shatter, or increase the risk for weather-related seed quality problems (Saskatchewan Pulse Growers, 2011). Such conditions necessitate the use of desiccants to defoliate and dry the crop down for harvesting operations.

## **2.4 Weed competition in lentil**

Weed control in lentil is a major issue for lentil producers (Brand et al., 2007; Yenish et al., 2009). Lentil is generally considered a poorly competitive crop against weeds because of its short height and poor early season vigor (Brand et al., 2007; Yenish et al., 2009). The yield reduction in lentil associated with weeds has been reported to be as high as 80% (Brand et al., 2007). Thus, it is important to manage weeds in lentil to maximize seed yield (Brand et al., 2007; Yenish et al., 2009). Although late emerging weeds have few adverse effects on the absorption of water, nutrients, and radiation, they play a significant role in decreasing seed quality, grain yield and harvestability (Gabe, 1994; Brand et al., 2007; Yenish et al., 2009). Late emerging weeds can decrease lentil seed grade because green weeds increase plant moisture content and dockage in the harvested seed (Gabe, 1994; Brand et al., 2007; Yenish et al., 2009; Saskatchewan Pulse Growers, 2011). Green weeds at harvest also can lower crop harvest operation efficiency (Muehlbauer et al., 2002; Brand et al., 2007; Yenish et al., 2009). For example, some perennial weeds like Canada thistle (*Cirsium arvense* L.) or dandelion (*Taraxacum officinale* L.) can make

swathing or straight cutting more difficult because of green stems sticking to the cutter bar (Muehlbauer et al., 2002; Saskatchewan Pulse Growers, 2011). Therefore, it is desirable to desiccate late emerging weeds prior to lentil harvest (Gabe, 1994; Brand et al., 2007; Yenish et al., 2009).

Traditionally, economic thresholds ignored the threat of late season weed seed production because they rarely reduce the yield of the cultivated crop (Bagavathiannan and Norsworthy, 2012). Late-season weed seeds are a source of seed bank replenishment, however, resulting in future weed problems and an increased potential for herbicide resistance (Bagavathiannan and Norsworthy, 2012; Norsworthy et al., 2012; Norsworthy et al., 2014). A new push in weed science towards a zero-tolerance threshold has recently garnered attention because it attempts to prevent all weeds that are prone to resistance from escaping control. Moreover, it is necessary for long-term weed control, especially for a weed species with excess seed production and rapid dispersal (Bagavathiannan and Norsworthy, 2012; Norsworthy et al., 2012; Norsworthy et al., 2014). Bagavathiannan and Norsworthy (2012) suggested that pre-harvest application of herbicides is an excellent way to manage late growing weed seed escapes due to the adverse effects of desiccants on weed seed production and vigor.

## **2.5 Pre-harvest desiccants**

In order to improve lentil seed yield, quality, and harvest efficiency, some pre-harvest treatments are used to desiccate crops quickly and uniformly (Riethmuller et al., 2005; Saskatchewan Pulse Growers, 2011; Schemenauer, 2011; Alberta Pulse Growers, 2013; Fleury, 2015). These also dry-down green weeds in the field, and some can provide weed control, especially for perennial weeds (Riethmuller et al., 2005; Ali et al., 2009; Saskatchewan Pulse Growers, 2011; Schemenauer, 2011; Alberta Pulse Growers, 201; Fleury, 2015). Therefore, chemical desiccation is a practical and popular harvest aid in lentil production. Chemical desiccation requires less time to dehydrate the lentil crop to a suitable seed moisture for harvest compared to swathing or natural dry-down (Tang et al., 1992). Herbicides applied as pre-harvest aids destroy the plant and prevent it from taking water or nutrients up from the soil (Tang et al., 1992). Riethmuller et al. (2005) reported that lentil yield under desiccated treatments was higher than under machine harvesting treatments that did not receive any desiccation. The authors

concluded that chemical desiccation is a rapid way to dry-down lentil crops. Chemical desiccation may exhibit greater performance in retaining seed yield and quality compared with swathings without any chemical desiccants, as the longer lentil is exposed to adverse environmental conditions, the greater the yield losses and seed quality reductions (Tang et al., 1992; Saskatchewan Pulse Growers, 2011).

The function of chemical desiccants for achieving successful harvest has been widely studied by a number of researchers in other crops. For instance, Ellis et al. (1998) conducted experiments to determine the influence of numerous herbicides (glyphosate, glufosinate, paraquat, oxyfluorfen, sodium chlorate and bromoxynil) as desiccants on yield and weed control in soybeans. They reported that most treated plots had similar yield to untreated plots after desiccant treatments were applied (Ellis et al., 1998). Likewise, desiccants applied to soybean at various application timings effectively accelerated soybean desiccation compared to untreated controls for both indeterminate and determinate soybean cultivars (Boudreaux and Griffin, 2011). Soltani et al. (2013) applied diquat, carfentrazone-ethyl, glufosinate, flumioxazin or saflufenacil alone or tank-mixed with glyphosate as desiccants to dry bean. None of the treatments influenced crop yield, and all but carfentrazone-ethyl provided consistent crop desiccation (Soltani et al., 2013). The benefits of desiccants in wheat (*Triticum aestivum* L.) were reported by Darwent et al. (1994). Their results showed significant reductions in wheat seed and foliage moisture contents following glyphosate application between 20% to 40% seed moisture content (Darwent et al., 1994). Similarly, Gubbels et al. (1993) found that desiccants applied to flax (*Linum usitatissimum* L.) desiccated leaves, capsules, and most stems more uniformly than the untreated control, but diquat and glufosinate-ammonium provided a shorter capsule dry-down period (about 1 week) compared with glyphosate (about 2 weeks). Research conducted on rice showed that desiccants accelerated crop desiccation by reducing harvest moisture content without yield loss when they were applied at the proper moisture content (Bond and Bollich, 2007). Collectively, these studies demonstrate the advantages of using desiccants for rapid and uniform crop dry-down, thereby allowing easier harvesting in numerous crops.

It has also been suggested that combinations of desiccants may have similar or improved effects on increasing harvest efficiency compared to those traditional products containing only one desiccant (Ellis et al., 1998; Bennett and Shaw, 2000a; Soltani et al., 2013). Ellis et al. (1998) evaluated the influence of herbicide combinations including paraquat, glyphosate,

oxyfluorfen and bromoxynil in combination with sodium chlorate. Their data showed that those herbicide combinations increased weed control without concomitant soybean yield losses (Ellis et al., 1998). Bennett and Shaw (2000b) also confirmed that herbicide combinations had good pre-harvest effects on the control of late emerging weeds in soybean. They observed that the application of paraquat tank-mixed sodium chlorate effectively reduced *Sesbania exaltata* (L.) seed growth in subsequent years by allowing less time for seed maturation (Bennett and Shaw, 2000b). A more recent study by Soltani et al. (2013) showed that diquat, carfentrazone-ethyl, glufosinate ammonium, flumioxazin, and saflufenacil tank-mixed with glyphosate facilitated dry bean desiccation and weed control.

In addition to improved crop dry-down and weed control, applying tank-mixtures of herbicides as desiccants is also helpful for managing herbicide resistance. This strategy can reduce selection pressure on resistant-prone weeds for some vulnerable herbicides (Wrubel and Gressel, 1994). Compared with applying herbicides individually, the combination of two or more herbicides with different modes of action may be useful to reduce the rate of resistance to both herbicides (Wrubel and Gressel, 1994).

## **2.6 Properties of chemical harvest aids**

Chemical desiccation products can be classified into two groups. One group is called a true desiccant, and consists of herbicides with contact action and rapid activity. For example, diquat (Reglone®) is a true desiccant because it rapidly desiccates plants with virtually no translocation in the plant (Schemenauer, 2011). The other group consists of pre-harvest aids or systemic herbicides with slower dry-down effects on the crop, such as glyphosate (Schemenauer, 2011).

In Canada, diquat (Group 22), which is highly toxic to mammals, is registered as a desiccant in lentils (Fleury, 2015; Saskatchewan Ministry of Agriculture, 2014). It is a non-selective, contact herbicide (Cobb and Reade, 2010; Saskatchewan Ministry of Agriculture, 2014). Diquat rapidly desiccates all plant tissue that the product contacts (Schemenauer, 2011). Although it can translocate in the xylem, translocation is limited by rapid desiccation. Thus, adequate coverage of the plant is important to achieve good desiccation (Saskatchewan Ministry of Agriculture, 2014). Diquat is stable under physiological pH values and binds to the soil tightly, so it exhibits no soil activity or soil residual problems (Cobb and Reade, 2010).

Diquat affects the electron transport chain of photosynthesis at Photosystem I, disrupting internal cell membranes by disrupting proteins and lipids (Fuerst and Norman, 1991; Cobb and Reade, 2010). More specifically, diquat works as a catalyst and diverts electrons from the electron carrier  $\text{FeS}_{\text{AB}}$ , which is a protein-bound iron-sulfur molecule that transports electrons to ferredoxin (Fuerst and Norman, 1991; Cobb and Reade, 2010). Those diverted electrons react with oxygen and form ultra-reactive hydroxyl radicals, which peroxidize proteins and lipids, resulting in rapid plant desiccation (Fuerst and Norman, 1991; Cobb and Reade, 2010). Typically, lentil can be harvested 7 to 10 days after treatment with diquat (Schemenaure et al., 2011; Alberta Pulse Growers, 2013; Saskatchewan Ministry of Agriculture, 2014), and although reduced sunlight will prolong the dry-down period, it will produce a more even, thorough dry-down due to increased (although still limited) translocation of the product. Diquat application is recommended when seeds have reached 30% moisture content (Saskatchewan Ministry of Agriculture, 2014).

Glufosinate is also registered as a desiccant for lentil in Canada (Fleury, 2015). It is a contact herbicide with low toxicity to mammals. Glufosinate is non-selective and can translocate in the phloem and xylem, but like diquat, movement is limited by its rapid activity. Glufosinate is a Group 10 product that exhibits activity as a glutamine synthetase inhibitor, binding to glutamine synthetase irreversibly and limiting the conversion of glutamate and ammonium into glutamine (Devine et al., 1993; Cox et al., 1996; Cobb and Reade, 2010). Plant death occurs from the accumulation of inorganic ammonium or glyoxylate, which inhibits RUBISCO and reduces the efficiency of photosynthesis (Devine et al., 1993; Cobb and Reade, 2010). Glufosinate should be applied to lentil when 40 to 60% of pods are turning brown (Saskatchewan Ministry of Agriculture, 2014).

Glyphosate was developed by the Monsanto Company and first marketed in 1974; today it is one of the most used herbicides in the world (Ashigh and Hall, 2010). Glyphosate is a non-selective herbicide and has low toxicity to mammals. Unlike diquat or glufosinate, which both have rapid, contact action, glyphosate is a broad-spectrum systemic herbicide that is translocated in the phloem and xylem and slowly inhibits plant growth (Devine et al., 1993; Cobb and Reade, 2010; Saskatchewan Ministry of Agriculture, 2014). Therefore, glyphosate is not a true desiccant, but has nevertheless been registered as a harvest aid in lentil. It is typically applied in pulse crops to control perennial weeds and to assist in crop dry-down (Schemenaure et al., 2011;

Alberta Pulse Growers, 2013). The unique mode of action of glyphosate involves the inhibition of 5-enolpyruvylshikimate-3-phosphate synthase (EPSPS), an important enzyme in the shikimate pathway used to produce aromatic amino acids in plants (Devine et al., 1993; Reddy et al., 2004; Duke and Powles, 2008; Ashigh and Hall, 2010; Cobb and Reade, 2010). Inhibition of EPSPS also leads to the accumulation of shikimate or shikimate-3-phosphate (Devine et al., 1993; Cobb and Reade, 2010). However, plant death by EPSPS inhibition is still not fully understood (Duke and Powles, 2008), but it is believed that plant death occurs from the starvation of aromatic amino acids and carbon, as well as from the accumulation of toxic intermediates such as shikimate or shikimate-3-phosphate (Duke and Powles, 2008). Glyphosate is typically applied to lentil when the lower 35% of the pods have turned brown (Schemenauer, 2011; Saskatchewan Ministry of Agriculture, 2014).

Recently, some relatively new desiccants have proven to be beneficial in enhancing lentil dry-down. Saflufenacil (Group 14), developed by BASF, can inhibit the protoporphyrinogen IX oxidase (PPO) enzyme, which converts protoporphyrinogen IX to protoporphyrin IX (Soltani et al., 2010; Ashigh and Hall, 2010; Grossmann et al., 2010). The inhibition of the PPO enzyme prevents biosynthesis of chlorophyll and heme (Matrige et al., 1992; Grossmann et al., 2010), ultimately leading to cell membrane destruction and necrosis (Duke et al., 1991; Grossman et al., 2010). Saflufenacil is a relatively new PPO inhibitor that is utilized in broadleaf weed control for small grains, and for desiccating crops such as sunflowers (*Helianthus* L.) (United State Environmental Protection Agency, 2009). Although normally applied to the plant foliage, this herbicide is absorbed by both the roots and leaves of plants (Soltani et al., 2010). Saflufenacil has mobility in both the phloem and xylem, but exhibits limited translocation in the phloem (Soltani et al., 2010). This property of saflufenacil is unique as most other Group 14 products have movement in the xylem only (Soltani et al., 2010). Saflufenacil has relatively low toxicity to mammals and has a short persistence in soil (United State Environmental Protection Agency, 2009; Soltani et al., 2010). Similar to glyphosate, the optimal timing for saflufenacil (when used as a harvest aid in lentil) is when 15% of bottom pods are mature and brown with ripened seeds inside (BASF, 2014).

Pyraflufen-ethyl and flumioxazin are also PPO-inhibitors that are classified as Group 14 products (Saskatchewan Ministry of Agriculture, 2014). Both are translocated in the xylem and both are used sporadically for dry-down. Pyraflufen-ethyl was primarily introduced to control



broadleaved weeds in cereal crops in 1993 (Miura, 2003), and was labeled as a harvest aid in cotton (*Gossypium hirsutum* L.) and potatoes (*Solanum tuberosum* L.) (Ivany, 2005; Nichino Europe Co. Limited, 2012). Pyraflufen-ethyl is a contact herbicide and has rapid foliar impacts on plants (Nichino Europe Co. Limited, 2012). As a desiccant for cotton and potato, its application timing should be the onset of natural senescence of potato, or achieving adequate cotton bolls (Nichino Europe Co. Limited, 2012).

Flumioxazin (Group 14), developed by Valent U.S.A., provides residual weed control and good desiccation of dry bean (Valent Canada, Inc., 2009; Soltani et al, 2013). It is a new option for controlling weeds that are resistant to Group 2 and 5 herbicides (Valent Canada, Inc., 2009). Flumioxazin is a contact herbicide that can provide control of many weeds including pigweeds (*Amaranthus palmeri* L.), common ragweed (*Ambrosia artemisiifolia* L.), dandelion, green foxtail (*Setaria viridis* L.), common lamb's quarters (*Chenopodium album* L.) (Valent Canada, Inc., 2009). It can dissipate in water and soil rapidly, resulting in low herbicide residue for crop rotations (Valent Canada, Inc., 2009). To date, there is no registration of flumioxazin for use as a lentil desiccant.

## **2.7 Limitations of chemical harvest aids**

Desiccants have been widely used as harvest aids by producers, but they may also have some limitations. First, improper application timing of these harvest aids may have adverse effects on crop seed yield and quality, resulting in economic losses (Fleury, 2015). Research on soybean has indicated there could be reductions in crop yield and quality if desiccants are used at an improper crop stage (Whigham and Stroller, 1979; Cerkauskas et al., 1982; Ratnayake and Shaw, 1992; Ellis et al., 1998; Bennett and Shaw, 2000a; Boudreaux and Griffin, 2011). Thus, the application timing of desiccants is critical. Boudreaux and Griffin (2011) reported that applying desiccants at 60% seed moisture content reduced the yield of indeterminate soybean cultivars by 15.4%, and decreased 100-seed weight by 12.4%. The authors reported that some of the soybean seeds at the top of the plant had not reached physiological maturity at the early application timing (about 60% seed moisture content); consequently, yield loss and reduced seed weights were observed compared to delayed applications (Boudreaux and Griffin, 2011). Similar results have been reported by Bennett and Shaw (2000a). Their results showed a decrease in soybean

yield, seed weight, and the subsequent germination, emergence and seedling growth of seedlings when desiccants were applied before the R7 (beginning of maturity) crop stage of soybean (Bennett and Shaw, 2000a). Apart from the research on soybean desiccation, application timing of desiccants on other crops has also demonstrated the importance of proper application timing. Moyer et al. (1996) noted that alfalfa was adequately desiccated by glufosinate without yield loss when approximately 60% of the pods had turned brown.

Accumulation of herbicide residues in crop seed is another important concern with the use of desiccants. The chemical residue of desiccation products can be detected in harvested seeds if they are applied too late in the seed development. In some cases, the active ingredient can be translocated into growing seeds, which increases the amount of residue that accumulates in the seed (Cessna et al., 1994, 2000; 2002). For example, the accumulation of glyphosate and its metabolites, aminomethylphosphonic acid (AMPA), may cause problems for Canadian lentil exports. Some countries have set their own maximum residue limits (MRLs) and thus, harvested seeds with levels exceeding the MRL cannot be exported to those countries. In order to avoid financial losses and trade restrictions due to MRLs in harvested seed, growers must ensure herbicide residues are below the MRL for any given crop. Herbicide residues can be influenced by herbicide rates, application timing, and the environmental conditions at the time of application (Cessna et al., 1994, 2000; 2002). Producers are recommended to follow the product label for appropriate application timing and use rate, and check MRLs of different international markets. Table 2.1 shows the various glyphosate and saflufenacil MRLs in lentils for the European Union, International standards, Japan, United States, and Canada (Bryant Christie Inc., 2015).

**Table 2.1** Maximum Residue Limits (MRLs) in parts per million (ppm) in lentil for European Union, International CODEX (International standards), Japan, United States and Canada. Adapted from Global MRL Database (Bryant Christie Inc., 2015).

Herbicide	Established MRLs				
	European Union	International codex	Japan	United States	Canada
			ppm		
Glyphosate	10.00	5.00	2.00	8.00	4.00
Saflufenacil	0.03	0.30	0.30	0.30	0.30

In some cases, certain desiccants may not be recommended depending on the crop and its potential end use. For example, products containing glyphosate are not recommended when lentil is grown for seed because glyphosate can cause low seed germination, vigor, and yield (Alberta Pulse grower, 2013). Yenish and Young (2000) found that there were adverse effects on germination rate, thousand seed weight, seedling density and height compared with untreated wheat when glyphosate was applied at the milk stage of wheat development. Similar results were shown in field pea, whereby the authors attempted to assess the effects of glyphosate on subsequent seedling emergence and vigor (Baig et al., 2003). This research suggested that glyphosate could not be recommend as a harvest aid if the crop was to be grown for seed because of seedling damage, including low seedling vigor and germination (Baig et al., 2003). These abnormalities may be caused by vascular tissue differentiation of the proembryo and immature embryo resulting from reduced accumulation of storage protein polypeptides (Shuma and Raju, 1993).

## **3.0 Impact of Glyphosate and Saflufenacil Application Timing on Lentil Seed Yield, Quality and Herbicide Residues**

### **3.1 Introduction**

Lentil is one of the most important pulse crops in western Canada, with production totals of 1.5 million tonnes in 2011 (Statistics Canada, 2014). Saskatchewan is the major lentil producer in Canada, accounting for 96% of the total harvested area (AAFC, 2013b). However, the indeterminate growth habit of lentil combined with variability in field conditions can result in non-uniform maturity (Saxena, 2009), which may decrease seed quality and slow harvesting operations (Saskatchewan Pulse Growers, 2011; Alberta Pulse Growers, 2013). Thus, growers typically desiccate the lentil crop once it reaches physiological maturity (Saskatchewan Pulse Growers, 2011; Schemenauer, 2011; Alberta Pulse Growers, 2013). Desiccating lentil can improve lentil dry down and control late-emerging weeds, which allows for early harvesting and enhances lentil harvest efficiency (Riethmuller et al., 2005; Ali et al., 2009; Saskatchewan Pulse Growers, 2011; Schemenauer, 2011; Alberta Pulse Growers, 2013).

Some herbicides are registered in Canada as desiccants to promote lentil desiccation. Examples include diquat, glyphosate, saflufenacil, and glufosinate (Saskatchewan Ministry of Agriculture, 2014). These herbicides are applied late in the growing season and consequently, herbicide residue can be detected in seeds and may cause trade issues if residue levels exceed the maximum residue limits (MRL) for importing countries. Maximum residue limits vary by crop, herbicide, and foreign market requirements (Bryant Christie Inc., 2015). Thus proper application timing of desiccants is critical for market acceptance (Cessna et al., 1994, 2000; 2002; Saskatchewan Pulse Growers, 2011), as improper application timing may reduce crop seed yield, seed weight, and seedling vigor (Bennett and Shaw, 2000a; Boudreaux and Griffin, 2011). Bennett and Shaw (2000a) reported that soybean yield, thousand seed weight, and seedling vigor decreased when desiccants (glyphosate, paraquat, and sodium chlorate) were applied before soybean reached maturity. Boudreaux and Griffin (2011) also found similar results, showing improper application timing (50% application seed moisture or higher application seed moisture) of desiccants led to a decrease in soybean seed yield and seed weight. Improper application timing of desiccants can also increase herbicide residues in crop seeds. Cessna et al. (1994, 2000;

2002) documented increased glyphosate residue with pre-harvest application of glyphosate when applied at earlier growth stages for spring wheat (*Triticum aestivum* L.), field pea (*Pisum sativum* L.), barley (*Hordeum vulgare* L.), and flax (*Linum usitatissimum* L.), and canola (*Brassica napus* L.).

Glyphosate is commonly used as a harvest aid in Canadian pulse and cereal crops. It provides good perennial grassy and broadleaf weed control, and may reduce the time between physiological maturity and harvest (Cessna et al., 2000; Cessna et al., 2002; Schemenauer et al., 2011). Glyphosate is absorbed via the foliage and translocates through the phloem to actively growing plant tissues (sucrose sinks) (Cessna et al., 1994, 2000; 2002; Duke and Powles, 2008). The recommended application timing is typically when the crop is at or below 30% seed moisture content (Saskatchewan Ministry of Agriculture, 2014). However, if glyphosate is applied to crops that have not reached physiological maturity, the herbicide may be translocated to developing seeds and accumulate there (Cessna et al., 1994, 2000; 2002). If glyphosate residue levels exceed the acceptable level (MRLs), some import markets may reject the seed shipment, resulting in economic loss for growers and reduced commerce for exporters.

Saflufenacil, a protoporphyrinogen IX oxidase inhibitor with rapid crop dry-down, has recently been introduced to the market and is newly registered as a desiccant in lentil (Soltani et al., 2009; Grossman et al., 2010). Saflufenacil has both contact and systemic activity via limited translocation through the phloem and xylem, and could translocate to sucrose sinks such as seeds. Therefore, saflufenacil residues also may be a concern for growers if it is applied as a desiccant at early crop stages. Similar to glyphosate, major importing countries also have set MRLs for saflufenacil (Bryant Christie Inc., 2015).

Saflufenacil provides more rapid weed control than glyphosate, but does not provide adequate control of perennial weeds in lentil (Baylis, 2000; Schemenauer, 2011). As a result, growers should apply both products if they are seeking rapid crop dry-down and perennial weed control. Several studies have evaluated the interactions between saflufenacil and glyphosate. Ashigh and Hall (2010) reported that the activity of glyphosate was reduced in plants when combined with saflufenacil. The authors attributed this to saflufenacil's rapid contact activity, which caused accelerated cell death and decreased the time allowed for glyphosate to be translocated to growing plant tissues (Ashigh and Hall, 2010). Meanwhile, saflufenacil translocation was reduced in glyphosate-susceptible plants by adding glyphosate (Ashigh and Hall, 2010).

However, Knezevic et al. (2009) reported that the mixture of saflufenacil and glyphosate improved the activity of both herbicides in controlling several weeds.

In 2011, the European Union rejected shipments of Canadian lentils due to glyphosate seed residues over 0.1ppm (Pratt, 2011). This had significant impacts on the Pulse industry in Canada, and it raised questions about effective control of glyphosate residue in lentil. Consequently, research was required to determine more effective timings of glyphosate and also, to assess whether new products, such as saflufenacil, could improve lentil desiccation if combined with glyphosate. Because glyphosate is preferred by producers to control perennial weeds in lentil crops, it is important to understand how lentil seed yield and size are affected by glyphosate timing. Moreover, the interaction between glyphosate and new products such as saflufenacil must be understood to determine its influence on crop yield and quality, as well as herbicide residue. Therefore, the objective of this research was to determine the response of lentil to various application timings of glyphosate, saflufenacil, and the combination of these two herbicides applied in a tank mix. A second objective was to determine whether the addition of saflufenacil to glyphosate at various application timings had any impact on herbicide residues in seeds.

### **3.2 Hypotheses**

The hypotheses of this study are that early application of glyphosate, saflufenacil, and a tank mix of glyphosate+saflufenacil will result in adverse effects on the crop and unacceptable herbicide residues. Secondly, tank-mixing glyphosate with saflufenacil will reduce levels of herbicide residue without adverse effects on lentil crops.

### **3.3 Materials and Methods**

#### **3.3.1 Experiment site and design**

A field trial was conducted at Saskatoon and Scott, Saskatchewan (SK), Canada, from 2012 to 2014. However, the trial at Scott in 2012 was lost due to hail damage and will not be discussed further. The soil texture at Saskatoon ranged from a clay to a sandy loam with a pH of 7.5 to 7.9

and an organic matter content of 2.4% to 4.5%. The soil at Scott has a silty loam texture with a pH of 5.3 to 6.8 and an organic matter content of 2.4% to 2.6%.

Plots were set up in a randomized complete block design with four replications per treatment. Two experimental factors were used in the study: herbicide treatment (glyphosate, saflufenacil and the tank mixture of glyphosate plus saflufenacil) and application timings (60%, 50%, 40%, 30%, and 20% seed moisture content). An unsprayed control also was included in the study. Individual plot sizes were 2 m wide by 6 m long and 2 m wide by 5 m long at Saskatoon and Scott, respectively.

### 3.3.2 Experimental procedure

CDC Maxim, the most widely grown small red lentil cultivar in western Canada, was selected for this trial. CDC Maxim is a high yielding cultivar with resistance to imidazolinone (group 2) herbicides (Saskatchewan Ministry of Agriculture, 2010). Prior to planting, seeds received a seed treatment consisting of Apron Maxx RTA (0.73% fludioxonil; 1.10% metalaxyl-M and S-isomer) at a rate of 325 ml per 100 kg seed. Seed was inoculated ( $2.76 \text{ ml kg}^{-1}$ ) with Liquid Nodulator® containing *Rhizobium leguminosarum* biovar *viceae* in 2012, or with Tag Team® Granular ( $2.8 \text{ kg ha}^{-1}$ ) containing *Rhizobium leguminosarum* and *Penicillium bilaii* in 2013 and 2014. Lentil was direct-seeded into chem-fallow plots at a depth of 3 cm. Planting was carried out with a small plot drill equipped with single shoot hoe openers on 22 cm row spacing. Planting dates at Saskatoon were May 17, 12, and 14 in 2012, 2013, and 2014, respectively; the Scott site was planted on May 21 and 12 in 2013 and 2014, respectively. Lentil target plant density was  $130 \text{ plants m}^{-2}$ , with seeding rates adjusted for germination test results. Plots were rolled at both sites immediately following planting to provide a smooth and level surface for harvest.

At Saskatoon, ethalfluralin was applied in the fall at a rate of  $1400 \text{ g a.i. ha}^{-1}$  to control weeds for the next year. An application of glyphosate ( $675 \text{ g a.e. ha}^{-1}$ ) was also made prior to crop emergence, while post-emergence weed control was achieved with a tank mix of imazamox plus imazethapyr ( $30 \text{ g a.i. ha}^{-1}$ ) applied between the 5<sup>th</sup> to 6<sup>th</sup> node stage. Any weeds not controlled by the herbicides were removed by hand. At the early flowering stage, prothioconazole was applied ( $166 \text{ g a.i. ha}^{-1}$ ) for the control of ascochyta blight, with a second application of

chlorothalonil applied ( $1500 \text{ g ha}^{-1}$ ) at the early pod stage if necessary. At Scott, imazethapyr was applied ( $13 \text{ g a.i. ha}^{-1}$ ) in the fall prior to plot establishment. Preemergence weed control was achieved with glyphosate ( $900 \text{ g a.e. ha}^{-1}$ ) applied immediately after planting, while an in-crop application of quizalofop-p-ethyl ( $420 \text{ g a.i. ha}^{-1}$ ) was made when the crop reached the 4<sup>th</sup> node. Preventative disease control was achieved with boscalid applied ( $294 \text{ g a.i. ha}^{-1}$ ) when lentil reached the early flowering stage.

Desiccants were foliar-applied as follows: glyphosate at  $900 \text{ g a.e. ha}^{-1}$ , saflufenacil at  $50 \text{ g a.i. ha}^{-1}$ , and glyphosate at  $900 \text{ g a.e. ha}^{-1}$  plus saflufenacil at  $36 \text{ g a.i. ha}^{-1}$ . All herbicides rates were based on label recommendations (Saskatchewan Ministry of Agriculture, 2014). Merge® adjuvant (50% surfactant and 50% petroleum hydrocarbons solvent) was added to treatments containing saflufenacil at a rate of 1 or  $0.5 \text{ L ha}^{-1}$  when applied alone or with glyphosate, respectively (Saskatchewan Ministry of Agriculture, 2014). Application timings and application dates are shown in Tables 3.1 and 3.2. Products were applied based on seed moisture content, with treatments being arranged in 10% seed moisture increments (60%, 50%, 40%, 30%, and 20%) to facilitate regression analysis. Herbicides were applied with a CO<sub>2</sub>-pressurized backpack sprayer (110-015 AirMix nozzle, 241 kpa, 45 cm spacing) at Saskatoon in 2012 and 2013 and with an air-pressurized tractor mounted sprayer equipped with shielding (110-015 AirMix nozzles, 275 kpa, 45 cm spacing) at Saskatoon in 2014. At Scott, a CO<sub>2</sub>-pressurized bicycle sprayer (110-003 AirMix nozzles, 276 kpa, 25cm) was used. All nozzles used to apply herbicides were calibrated to deliver  $200 \text{ L ha}^{-1}$  of spray water volume.



**Table 3.1** Herbicide treatments, rates, and application timings (% seed moisture content) for each herbicide treatment evaluated at Saskatoon and Scott, SK from 2012 to 2014.

Herbicide	Rate (g a.e. ha <sup>-1</sup> /g a.i. ha <sup>-1</sup> )	Application Timing (%)
Control		
Glyphosate	900	60 50 40 30 20
Saflufenacil§	50	60 50 40 30 20
Glyphosate+Saflufenacil†	900 + 36	60 50 40 30 20

§ A surfactant/solvent (Merge®) at 1 L ha<sup>-1</sup> was added in saflufenacil treatment.

† A surfactant/solvent (Merge®) at 0.5 L ha<sup>-1</sup> was added in the tank mixture of glyphosate+saflufenacil treatment.

**Table 3.2** Dates of application timings and environmental conditions (temperature, relative humidity and wind) for each herbicide treatment in timing trials at Saskatoon and Scott, SK from 2012 to 2014.

Site	Year	Application Timing	Application Date	Temperature	Relative Humidity
		(%)		(°C)	(%)
Saskatoon	2012	60	August 17	26.0	43.1
		50	August 20	29.0	33.0
		40	August 28	27.0	43.0
		30	August 30	16.0	55.0
		20	September 6	20.0	49.0
	2013	60	August 9	20.1	56.1
		50	August 14	20.3	69.0
		40	August 16	27.0	64.1
		30	August 19	30.1	30.5
		20	August 23	19.5	63.0
	2014	60	August 12	30.0	29.0
		50	August 15	24.0	66.7
		40	August 19	29.0	51.0
		30	August 27	30.0	35.5
		20	September 5	15.0	58.6
Scott	2012	NA	NA	NA	NA
	2013	60	August 20	13.4	73.9
		50	August 23	17.0	50.1
		40	August 29	19.6	74.5
		30	September 3	12.2	83.8
		20	September 12	10.7	61.8
	2014	60	August 12	19.1	70.8
		50	August 15	22.5	73.8
		40	August 19	20.4	69.3
		30	August 22	13.8	46.9
		20	August 27	21.0	49.3

NA: no applicable data recorded due to hail damage.

Prior to the application of harvest aid treatments, a random subsample of plants (10 plants per plot) was excised from border plots and bulked to create a composite seed sample on which seed moisture content could be determined. Each composite seed sample was weighed (fresh weight), placed in paper bags and dried in an oven at 80°C for 24 h to determine dry weight. Seed moisture content (SMC) of each sample was calculated by the following equation:

$$SMC = \frac{(M_f) - (M_d)}{M_f} * 100\% \quad [3.1]$$

where  $M_f$  is fresh weight of the composite seed samples, and  $M_d$  is the dry weight of the composite seed samples.

### 3.3.3 Data collection

Plant stand counts were performed two weeks after lentil emergence in two randomly selected, 1 m rows per plot. Desiccation was rated 7, 14, and 21 days after each herbicide application (DAA) based on the Canadian Weed Science Society visual scale (0 to 100%). The three visual ratings at 7, 14 and 21 DAA for each treatment were used to determine desiccation progress over time, which is calculated by the area under the desiccation progress curve (AUDPC):

$$AUDPC = \left(\frac{D_1 + D_2}{2}\right)(t_2 - t_1) + \left(\frac{D_2 + D_3}{2}\right)(t_3 - t_2) \quad [3.2]$$

where  $D_1$ ,  $D_2$ , and  $D_3$  represent observed desiccation ratings at each evaluation day;  $t_1$ ,  $t_2$ , and  $t_3$  represent the number of the days after each herbicide application (Jeger and Viljanen-Rollinson, 2001; Simko and Piepho, 2012). The AUDPC equation was used to convert the three desiccation ratings and crop moisture contents into a single relative value for the purpose of reporting; the greater the calculated AUDPC value, the further desiccation had progressed between ratings (McNaughton et al., 2015).

Lentil plots were harvested with a small plot combine at all sites except for the Saskatoon (2013) site, where a harvest error precluded data being collected for the 20% moisture content treatment. Harvested seeds were weighed (dirty weight), cleaned with a dockage tester, and weighed again to determine clean seed yield. Final yield was determined by calculating clean yield and then adjusting to 13% seed moisture content, which is the standard lentil seed moisture for storage. Thousand seed weight (TSW) was determined by counting and weighing 250 seeds and multiplying by a factor of four. Harvest straw moisture was tested immediately after harvesting each plot to determine if straw moisture will affect harvest efficiency. Fresh seed samples and plant straw were weighed (fresh weight), put into paper bags, oven-dried for 24 hours at 80°C and then reweighed.

To assess glyphosate residue levels at Saskatoon (2012 and 2013) and Scott (2013), samples of each treatment containing glyphosate and an untreated control were tested for glyphosate and AMPA residues. Each 250 g sample was collected at 7 DAA from border rows, cleaned, placed into plastic bags and kept in a freezer at -20°C until all samples were collected. Samples were sent to ALS laboratory in Edmonton, AB, Canada. Using a standardized process provided by

ALS Laboratories, high performance liquid chromatography (HPLC) using column switching and post-column derivatization with fluorescence detection was employed to determine glyphosate and AMPA residue. Briefly, a mixture of 150 ml of 0.1M hydrochloric acid and 50 ml of dichloromethane was added to ground samples. The solution was homogenized for 1 minute with a polytron, and centrifuged at 5000 RPM for 10 minutes. The aqueous layer of this solution (100 ml) was decanted to a flask and diluted with deionized water to 350 ml, and eluted through a Chelex 100 resin column at 2 drops per second. The wall of this column was then washed with 50 ml of deionized water and 100 ml of 0.2 M hydrochloric acid. All the eluent was discarded. Following this, 7 ml of 6 M hydrochloric acid was added to the column, and the eluent was discarded. 25 ml of 6 M hydrochloric acid was added again to the column, and with the eluent collected, mixed with 11 ml of concentrated hydrochloric acid and applied to a AG1-X8 resin column to remove excess iron. After the eluent entered the AG1-X8 resin column, the column was rinsed with 10 ml of 6 M hydrochloric acid, and the eluent was concentrated on a rotary evaporator. The extract of glyphosate and AMPA was then determined with an HPLC equipped with a fluorescence detector. Differential retention time was used to distinguish between glyphosate and AMPA, with a limit of detection of 0.020 ppm for both compounds.

Saflufenacil residue data was collected for both Saskatoon (2012, 2013, and 2014) and Scott (2013 and 2014) locations. Cleaned seed samples (75 g) were collected at 21 DAA, dried at 14°C in a paper bag, and then kept in freezer at -20°C until processed. Liquid chromatography-mass spectrometry (LC-MS) was used to determine the saflufenacil residues as per Mueller et al. (2014). Briefly, lentil seeds (5 g) were ground three times (15 seconds each duration) with a small grinder. Methanol (15 mL) was added and samples were shook for 1 hour. The samples and tubes were centrifuged at 3000 RPM for 1 minute, and filtered through a 0.45-micron filter directly to a 1.8 mL LC vial. The final solution was analyzed on the LC-MS system. Saflufenacil concentrations were determined by comparison to standards of known concentration responses. Saflufenacil recoveries were > 97% based on fortified untreated samples, so concentrations were not corrected for percent recovery (data not shown). The lower limit of detection of this procedure was  $5.6 \times 10^{-4}$  ppm of lentil seeds; all saflufenacil-treated samples had detectable saflufenacil residues.

### **3.3.4 Statistical analysis**

Residuals were initially tested for normality and homogeneity of variances with PROC UNIVARIATE and Levene's test, respectively (SAS Inst., 2014). Where residuals did not conform to the assumptions of ANOVA, heterogeneous variance structures were modeled with mixed models. All data were analyzed using the MIXED Procedure in SAS 9.3 (SAS Inst., 2014). Herbicide treatments and application timings were considered fixed effects in the model, while site-year (environmental effects), replication (nested within site-year), and the interaction between fixed and environmental effects were treated as random effects.

The significance of random effects and their interactions with fixed effects was assessed with the COVTEST option in PROC MIXED (SAS Inst., 2014). Meanwhile, scatterplots of variables were observed to determine whether data could be combined for analysis. Where data could not be combined, data were analyzed within site-years. Means were separated using Tukey's HSD test at  $P < 0.05$ . Single degree of freedom contrasts were used to make specific comparisons of interest. Where ANOVA indicated a significant effect of application timing, data were subjected to linear and quadratic regression analysis using PROC REG (SAS Inst., 2014).

## **3.4 Results and Discussion**

### **3.4.1 Lentil desiccation**

The interaction of site-year x application timing was significant for desiccation progress (Table 3.3) and thus, data were analyzed within site-years. The herbicide x application timing interaction did not significantly affect crop desiccation except at Saskatoon in 2012 and, thus desiccation data were combined across herbicide treatments for the other four site-years (Table 3.4). A significant regression between desiccation and application timing was only observed at Saskatoon in 2013 (Figure 3.1). Lentil desiccation decreased linearly with earlier application timing, with the least desiccation at 60% seed moisture content (Figure 3.1). A similar pattern was observed on dry bean by McNaughton et al. (2015), who reported that desiccation was consistently reduced at earlier crop growth stages. The authors suggested that plots with earlier

**Table 3.3** P-values derived from analysis of variance for area under desiccation progress curve (AUDPC), lentil seed yield, thousand seed weight (TSW), straw moisture, glyphosate residue (GR), and saflufenacil residue (SR) as influenced by herbicide (H) and application timing (T) at Saskatoon and Scott, SK from 2012 to 2014.

Source	AUDPC	Yield	TSW	Straw moisture	GR	SR
	P value					
Site-year (SY)	0.0935	0.11118	0.1045	0.1224	0.2509	0.4379
Timing (T)	0.8063	0.0826	0.2557	0.0037**	0.0184*	0.0002***
Herbicide (H)	0.0449*	0.7249	0.7054	0.1093	0.2397	0.0665
T x H	0.0029***	0.0032**	0.0449*	0.0017**	0.3670	0.0793
SY x T	0.0054**	0.0301*	0.0458	0.0208*	0.0505	0.1095
SY x H	0.0677	0.0395*	0.3877	0.0865	0.2444	0.1496
SY x T x H	0.4221	0.2016	0.0553	0.251	0.2188	0.0231*

\*, \*\*,\*\*\*, significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

**Table 3.4** P-values derived from analysis of variance illustrating fixed effects (herbicide and application timing) for the area under desiccation progress curve (AUDPC) at Saskatoon and Scott, SK from 2012 to 2014.

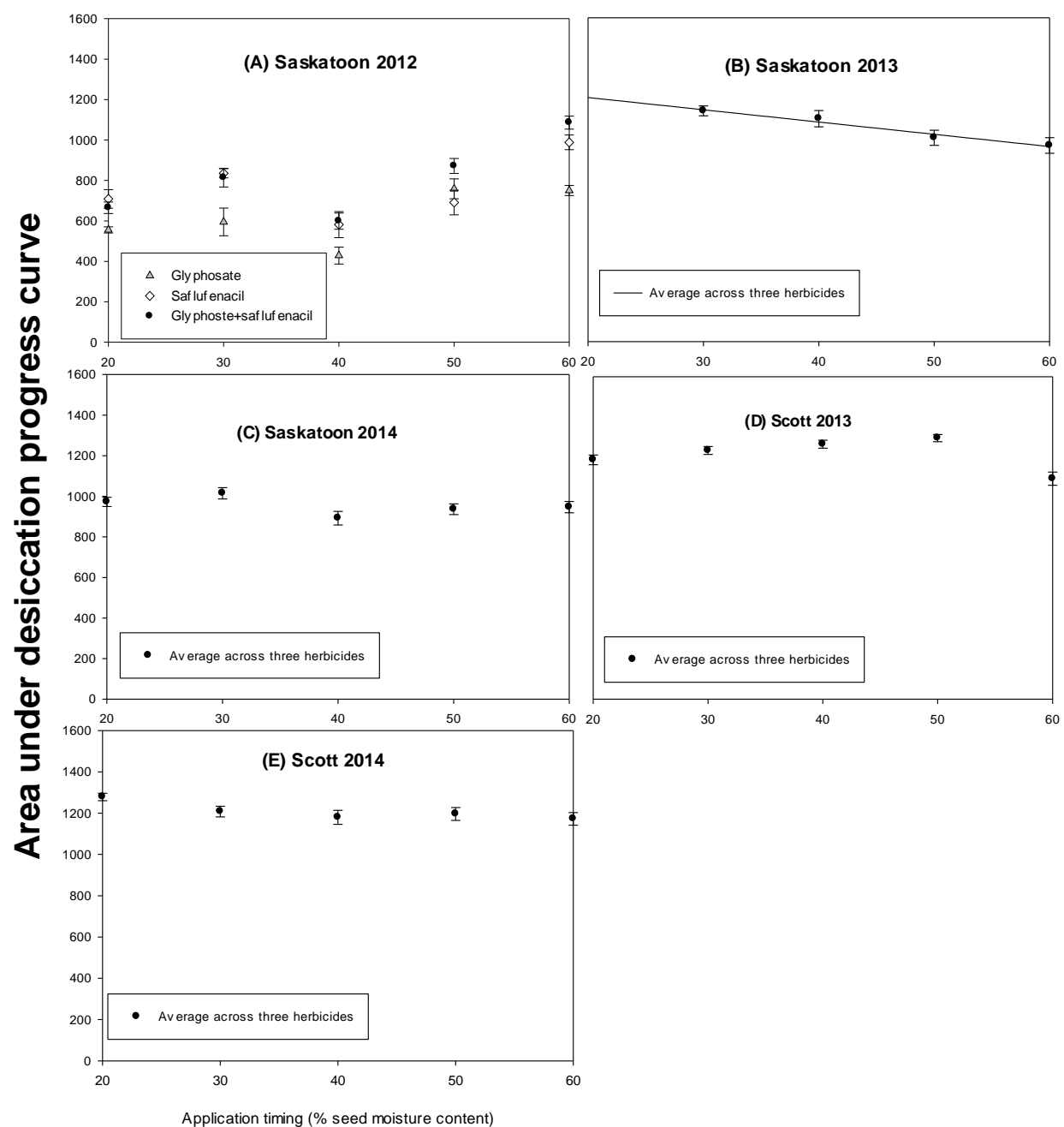
Source	AUDPC				
	P value				
	Saskatoon 2012	Saskatoon 2013	Saskatoon 2014	Scott 2013	Scott 2014
Herbicide (H)	<.0001***	0.2471	0.0030**	0.2791	0.3681
Timing (T)	<.0001***	0.0007***	0.0156*	<.0001***	0.0767
H x T	0.0098**	0.3928	0.1881	0.1450	0.3275

\*, \*\*,\*\*\*, significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

application timings had less time to desiccate naturally than plots receiving an delayed application timing (McNaughton et al., 2015). On the other hand, there were no statistically significant relationships detected in the current study across most of the site-years (Saskatoon 2012, Saskatoon 2014, Scott 2013, and Scott 2014). As Figure 3.1 shows, crop desiccation fluctuated with application timings at these site-years, leading to a lack of linear or quadratic responses (Figure 3.1).

Across five application timings, the contrasts showed that saflufenacil alone, or mixed with glyphosate, had faster desiccation than glyphosate alone at Saskatoon in 2012 (Table 3.5). Likewise, glyphosate+saflufenacil improved desiccation compared with each herbicide applied alone at Saskatoon in 2014 (Table 3.5). Glyphosate is a systemic herbicide that is an excellent harvest aid for weed control, but preforms poorly at crop desiccation (Schemenauer, 2011; Saskatchewan Ministry of Agriculture, 2014). However, saflufenacil works more rapidly within plants and requires less time to cause crop damage compared with glyphosate alone (Schemenauer, 2011). Our results corroborate those of Soltani et al. (2013) and McNaughton et al. (2015), who documented that the addition of saflufenacil to glyphosate increased dry bean desiccation compared to each herbicide applied alone. Knezevic et al. (2009) also reported that the mixture of saflufenacil+glyphosate increased glyphosate activity on dandelion. However, for several site-years (Saskatoon 2013, Scott 2013 and Scott 2014), our data showed no differences in crop desiccation between plots treated with glyphosate, saflufenacil, or their tank mixture (Table 3.5).

Generally, crop desiccation varied between site-years, potentially due to different variables such as temperature, relative humidity or soil properties (Table 3.2). For the site-years that had no significant patterns with delayed application timing, low temperature and rain occurred at later application timings (Table 3.2). This might lead to slower crop desiccation compared to earlier application timings, thereby minimizing the timing effects. A study by Willson and Smith (2002) concluded that harvest environments might impact the effects of glyphosate, glufosinate, and paraquat on desiccating dry bean. Wetter and cooler conditions resulted in slower crop maturation and reduced desiccation efficiency (Willson and Smith, 2002). Moyer et al. (1996) also reported that higher temperature and reduced rainfall resulted in faster alfalfa (*Medicago sativa* L.) desiccation.



**Figure 3.1** Relationship between area under desiccation progress curve (AUDPC) and application timing (% seed moisture content) at (A) Saskatoon 2012, (B) Saskatoon 2013, (C) Saskatoon 2014, (D) Scott 2013, and (E) Scott 2014. Regression equation across three herbicide treatments at (B) Saskatoon 2013:  $Y = -6.06x + 1327.70$ ,  $R^2 = 0.967$ ,  $P = 0.0167$ . No relationship was observed for each herbicide at any other site-year. Error bars represent one standard error of the mean.



**Table 3.5** Contrast statements of area under desiccation progress curve (AUDPC) represent comparisons for each herbicide treatment at various application timings (% seed moisture content), showing the estimate of difference between means at Saskatoon and Scott, SK from 2012 to 2014.

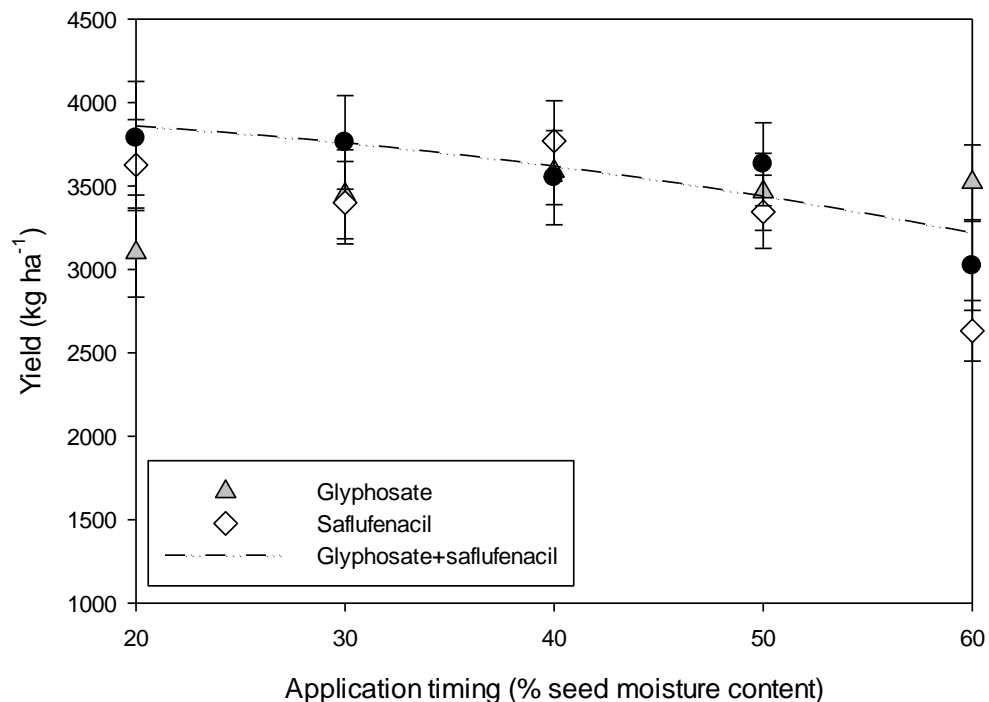
Herbicide compared	AUDPC				
	Saskatoon 2012	Saskatoon 2013	Saskatoon 2014	Scott 2013	Scott 2014
Glyphosate vs. Saflufenacil	-144***	6	-30	13	-19
Glyphosate vs. Glyphosate+saflufenacil	-190***	-51	-95***	-20	-40
Saflufenacil vs. Glyphosate+saflufenacil	-46	-57	-66*	-34	-22

\*, \*\*, \*\*\* , significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

### 3.4.2 Seed yield

Although the effects of site-year  $\times$  timing ( $P= 0.0161$ ), and site-year  $\times$  herbicide ( $P= 0.0382$ ) were statistically significant (Table 3.3), scatterplots of seed yield data from each site-year indicated that they had similar patterns. Moreover, the interactions of site-year  $\times$  application timing or site-year  $\times$  herbicide occupied relatively small proportions of the total sum of squares (5% and 9%, respectively) and showed little influence on model performance. Thus, seed yield data were pooled across site-years.

Lentil seed yield was affected by the interaction between herbicide treatment and application timing (Table 3.3) and so data were analyzed within herbicide treatments. Glyphosate alone did not affect lentil yield, regardless of application timing (Figure 3.2). Similar effects were observed for saflufenacil applied alone across all application timings, with the exception of 60% seed moisture content, where yield decreased ( $P< 0.05$ ) by 22% compared to untreated control (Figure 3.2). Lentil yield also decreased significantly when glyphosate was tank mixed with saflufenacil at earlier application timings (Figure 3.2). In fact, lentil yields were 25% greater when the tank mixture of glyphosate+saflufenacil was applied at 20% seed moisture compared with 60% seed moisture (Figure 3.2). In comparison to the untreated control, glyphosate+saflufenacil did not reduce yield at any of the application timings (Figure 3.2). The contrasts illustrated that there were no significant differences between the untreated control and the average of the three herbicides across application timings, which indicates that using desiccants did not result in lower seed yield than the untreated control (Table 3.6).



**Figure 3.2** Relationship between seed yield and application timing (% seed moisture content) across five site-years in Saskatchewan. Regression equation for the tank mixture of glyphosate+saflufenacil:  $Y = -0.2x^2 + 3940.4$ ,  $R^2 = 0.7935$ ,  $P = 0.0426$ . No relationship was observed for glyphosate or saflufenacil applied alone. Points ( $\blacktriangle$ ) represent glyphosate; points ( $\diamond$ ) represent saflufenacil; points ( $\bullet$ ) represent glyphosate+saflufenacil. Control yield was  $3358.0 \pm 252.0$  kg ha<sup>-1</sup>. Error bars represent one standard error of the mean.

**Table 3.6** Contrast statements of yield, thousand seed weight (TSW), and straw moisture, represent comparisons for each herbicide treatment at various application timings (% seed moisture content), showing the estimate of difference between means at Saskatoon and Scott, SK from 2012 to 2014.

Herbicide compared	Yield (Kg ha <sup>-1</sup> )	TSW (g)	Straw moisture (%)
Control vs. Glyphosate	-66.6	1.9*	5.3*
Control vs. Saflufenacil	10.9	2.1**	2.0
Control vs. Glyphosate+saflufenacil	-207.3	2.1**	5.8*
Glyphosate vs. Saflufenacil	77.5	0.3	-3.2*
Glyphosate vs. Glyphosate+saflufenacil	-140.7	0.2	0.6
Saflufenacil vs. Glyphosate+saflufenacil	-218.2	0.0	3.8*

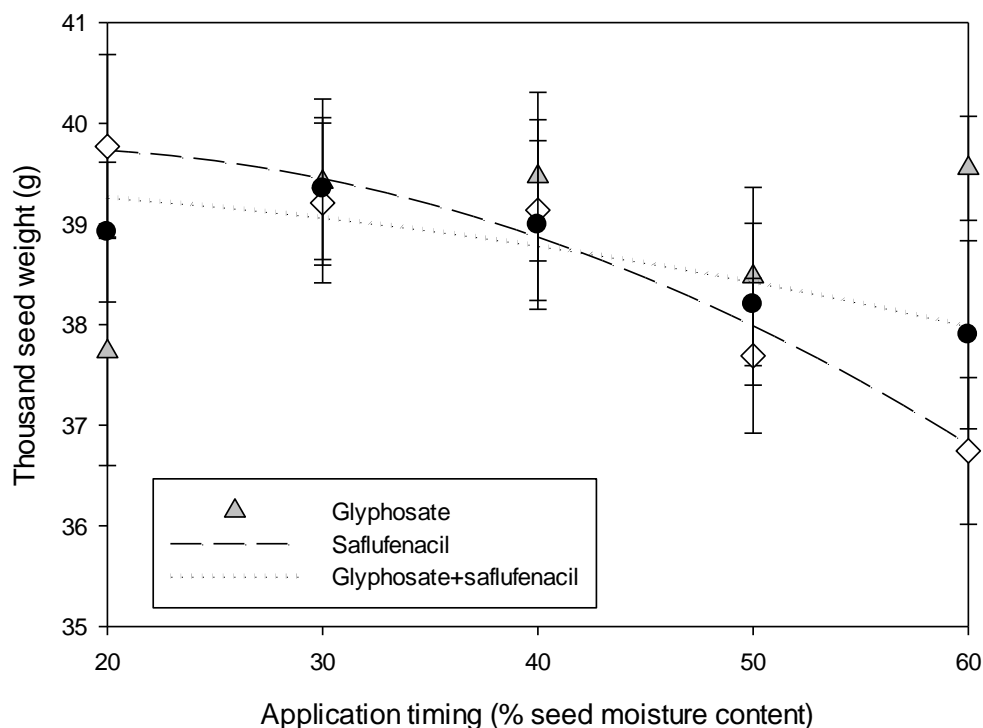
\*, \*\*,\*\*\*, significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

The results of the current study showed that lentil seed yield was generally reduced by an early application of the saflufenacil treatment. However, seed yield was not adversely influenced by herbicide treatments when applications were made at or below 50% moisture content (Figure 3.2). It is likely that at early application timings the lentil pods may have not reached physiological maturity. Similar results have been reported in rice (*Oryza sativa* L.), soybean (*Glycine max* L.), and dry bean (*Phaseolus vulgaris* L.). For example, Bond and Bollich (2007) showed that earlier applications of paraquat and sodium chorate (7 days before harvest) significantly decreased rice yield. Boudreaux and Griffin (2011) documented soybean yield reductions when harvest aid applications were made at the 50% and 60% seed moisture contents, but applications made at or later than the 40% seed moisture content stage did not have adverse effects on seed yield. McNaughton et al. (2015) found that glyphosate or saflufenacil alone or in a tank mixture increased soybean yield as applications were delayed to lower seed moisture contents. Results from our study indicate that 50% seed moisture content is earliest that applications could safely be made to the crop without compromising yield.

### **3.4.3 Thousand seed weight**

The interactions between site-year, herbicide treatment and application timing with respect to TSW were not significant therefore, TSW data were combined across site-years (Table 3.3). Due to an interaction between herbicide treatment and application timing, TSW data were analyzed within herbicide treatments (Table 3.3). There was no significant relationship between TSW and application timing when glyphosate was applied alone (Figure 3.3). However, quadratic responses were observed for both saflufenacil and the tank mixture of glyphosate+saflufenacil (Figure 3.3). TSW decreased from 39.8 g when saflufenacil was applied at 20% seed moisture content to 36.8 g when it was applied at 60% seed moisture (Figure 3.3). However, compared to the untreated control, there was no reduction in TSW with saflufenacil application until it was applied at 60% seed moisture content (Figure 3.3). Similarly, the tank mixture treatment of glyphosate+saflufenacil exhibited a curvilinear relationship with seed moisture content (Figure 2.3). Thousand seed weight decreased as moisture content increased down to a minimum of 37.7 g at 60% seed moisture content, but it was not statistically reduced compared to the untreated

control (Figure 3.3). Contrasts showed that all three herbicide treatments significantly ( $P<0.05$ ) reduced TSW compared with the untreated control (Table 3.6).



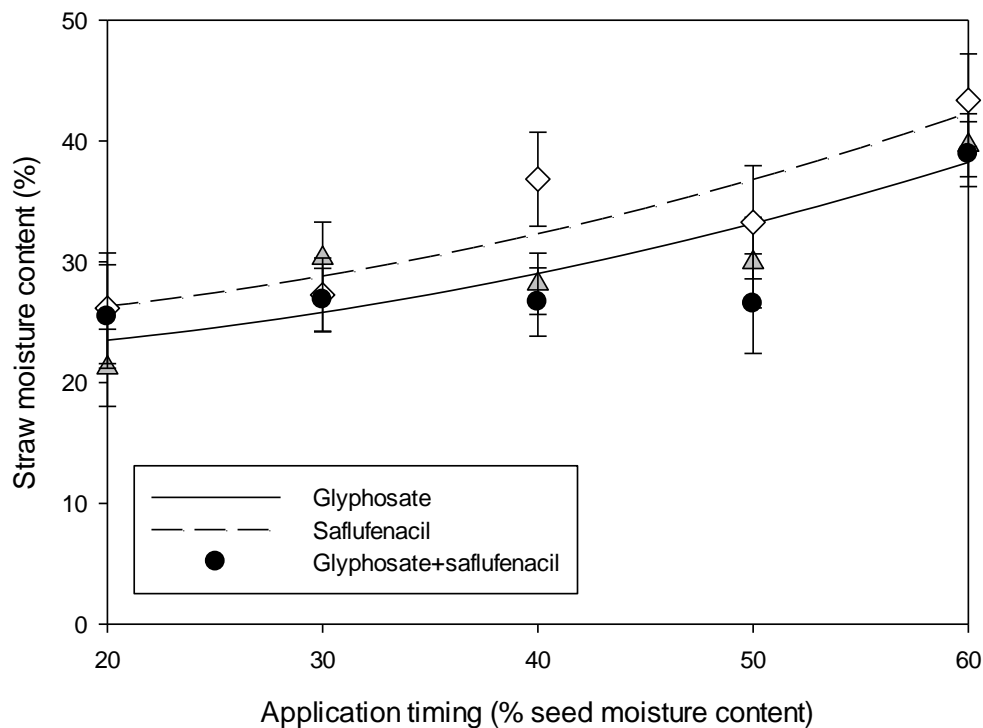
**Figure 3.3** Relationship between thousand seed weight and application timing (% seed moisture content) across five site-years in Saskatchewan. Regression equation for saflufenacil:  $Y = -0.0015x^2 + 0.0469x + 39.3940$ ,  $R^2 = 0.9680$ ,  $P = 0.032$ ; regression equation for the tank mixture of glyphosate+saflufenacil:  $Y = -0.0004x^2 + 39.4278$ ,  $R^2 = 0.7969$ ,  $P = 0.0415$ . No relationship was observed between TSW and glyphosate applied alone. Points ( $\blacktriangle$ ) represent glyphosate; points ( $\diamond$ ) represent saflufenacil; points ( $\bullet$ ) represent glyphosate+saflufenacil. Control TSW was  $40.6 \pm 0.8$  g. Error bars represent one standard error of the mean.

Based on the results of this study, there was no effect of application timing of glyphosate when it was applied alone (Figure 3.3). This agrees with the findings of Ratnayake and Shaw (1992) who observed that glyphosate did not affect soybean 100-seed weight if applied between the R5 (beginning seed development) and R8 (full seed maturity) growth stages. Saflufenacil treatments, on the other hand, produced a significant decrease in TSW when applications were made beyond 50% seed moisture content, which corresponds well with previous findings. A study by McNaughton et al. (2015) reported that dry bean seed weight consistently decreased when saflufenacil treatments were applied at earlier crop growth stages. Bennett and Shaw

(2000a) and Griffin and Boundreaux (2011) both found that there were significant reductions in soybean seed weight when desiccants were applied prior to 40% seed moisture content. The difference between glyphosate and saflufenacil observed in our study might result from the slow action of glyphosate at early growth stages, which permitted more time for seed growth prior to the arresting of seed development. In contrast, it is possible that saflufenacil rapidly limited lentil growth, which resulted in less time for seed development and lower seed weights. Although the impact of application timing of glyphosate was not obvious in this study, saflufenacil treatments displayed adverse effects on TSW at 60% seed moisture content. Therefore, growers must follow the application stages recommended on the product labels and avoid early application of these desiccants. In addition, applying the tank mixture treatment is an alternative because it did not show adverse effects on TSW compared with each herbicide alone.

#### **3.4.4 Harvest straw moisture content**

Harvested straw moisture data were combined across site-years. ANOVA indicated that the interaction of herbicide x timing significantly affected harvested straw moisture content and thus, data were analyzed within herbicide treatments (Table 3.3). For glyphosate and saflufenacil alone, harvested straw moisture content increased with early application timing, although the magnitude of the increase depended on herbicide treatments (Figure 3.4). For example, straw moisture content in the saflufenacil alone treatment increased nearly 50% from latest to the earliest application timing, whereas the glyphosate alone treatment exhibited an increase of 56% over the same treatments (Figure 3.4). Although there was no significant linear or quadratic relationship for the tank mixture of glyphosate+saflufenacil, straw moisture content was reduced compared to the untreated control, except at 60% seed moisture content (Figure 3.4). Contrasts indicated that there was no significant difference between the saflufenacil alone treatment and the untreated control (Table 3.6). However, all treatments containing glyphosate exhibited a significantly lower straw moisture content compared with the untreated control (Table 3.6).



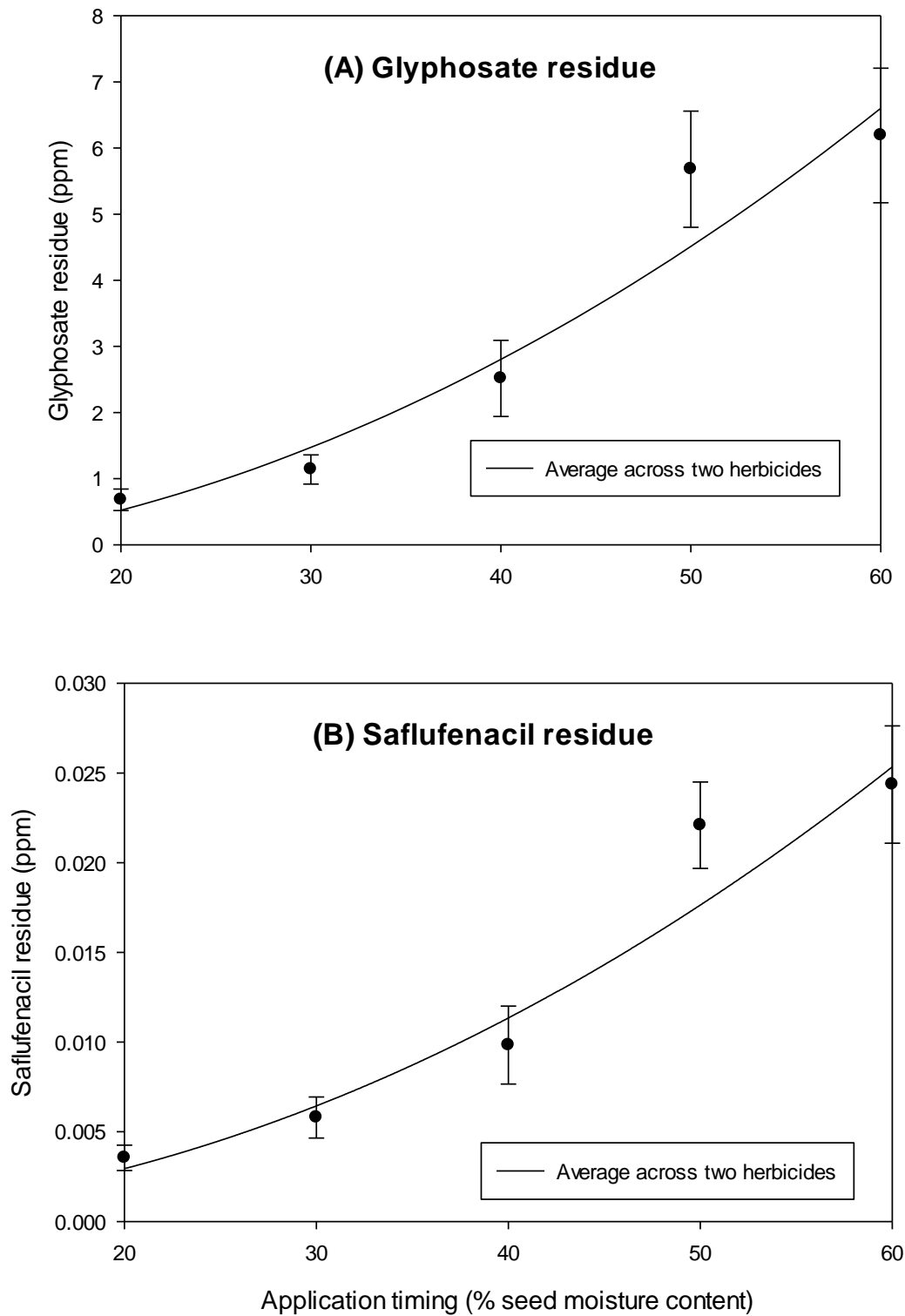
**Figure 3.4** Relationship between harvested straw moisture content and application timing (% seed moisture content) across five site-years in Saskatchewan. Regression equation for glyphosate:  $Y=0.0046x^2+21.6778$ ,  $R^2=0.7782$ ,  $P=0.0477$ ; regression equation for saflufenacil:  $Y=0.0050x^2+24.3442$ ,  $R^2=0.8185$ ,  $P=0.0348$ . No significant relationship was observed between the tank mixture of glyphosate+saflufenacil and straw moisture. Points ( $\blacktriangle$ ) represent glyphosate; points ( $\diamond$ ) represent saflufenacil; points ( $\bullet$ ) represent glyphosate+saflufenacil. Control straw moisture was  $34.8 \pm 4.1\%$ . Error bars represent one standard error of the mean.

High straw moisture content at harvest may reduce combine efficiency and slow combine speed. As expected, straw moisture content at harvest decreased with delayed application timing (Figure 3.4). When application timing is delayed, the crop experiences a longer desiccation period than for earlier application timings. This permits the crop to desiccate naturally in addition to the accelerated desiccation with the desiccants, ultimately resulting in better crop desiccation and lower straw moisture content (McNaughton et al., 2015). Compared with the untreated control, saflufenacil alone did not significantly improve straw desiccation, but glyphosate treatments did (Figure 3.4). This is a function of the rapid desiccation and contact-like action of saflufenacil, which has little-to-no translocation in the plant (Schemenauer, 2011). Taken together, the results indicate that regardless of the herbicide used, delayed application of desiccants will be more effective both in terms of crop desiccation and improved harvest efficiency due to lower straw moisture contents.

### 3.4.5 Herbicide residues

Glyphosate residue data were combined across site-years ( $n=3$ ) due to a lack of interactions between fixed and random effects (Table 3.3). Glyphosate residue data were not affected by the interaction between herbicide treatment and application timing ( $P= 0.3670$ ) and thus, data were pooled across herbicide treatments. Glyphosate residue increased from 0.7 at 20% seed moisture content to 6.2 ppm at 60% (Figure 3.5). This represents an approximate 8-fold increase in glyphosate residues at the earliest application timing compared with the recommended desiccation timing of 30% seed moisture content or less (Figure 3.5). Contrasts showed that adding saflufenacil to glyphosate did not influence glyphosate residues at any of the application timings (Table 3.7).

Although there was a significant site-year x timing x herbicide interaction, further examination of the data and residuals indicated that saflufenacil residue responded similarly to treatments across all site-years. Therefore, the data were pooled across years. Because saflufenacil residues were not affected by the interaction of herbicide treatment x application timing, data were combined across herbicide treatments (Table 3.3). Saflufenacil residues consistently decreased as the application timing was progressively delayed (Figure 3.5). For example, saflufenacil residue levels decreased approximately 85% as application timing was delayed from 60% to 20% seed moisture content (Figure 3.5). Contrasts showed that tank-mixing saflufenacil and glyphosate decreased saflufenacil residues compared to saflufenacil applied alone (Table 3.7). This is not surprising given that as per label recommendations, only two third of the rate of saflufenacil was used in the tank-mixture compared with when saflufenacil was applied alone (Table 3.1).



**Figure 3.5** Relationship between herbicide residue and application timing (% seed moisture content) across three site-years in Saskatchewan. (A) Regression equation of glyphosate residue across two herbicides:  $Y=0.0019x^2-0.2377$ ,  $R^2= 0.9339$ ,  $P= 0.0074$ . (B) Regression equation of saflufenacil residue across two herbicides:  $Y=0.0000072x^2+0.0001433$ ,  $R^2= 0.9372$ ,  $P= 0.0068$ . Error bars represent one standard error of the mean.



**Table 3.7** Contrast statements of glyphosate residue (GR) and saflufenacil residue (SR), represent comparisons for each herbicide treatment at various application timings (% seed moisture content), showing the estimate of difference between means at Saskatoon and Scott, SK from 2012 to 2014.

Herbicide compared	GR	SR
	(ppm)	
Glyphosate vs. Glyphosate+saflufenacil	1.1	NA
Saflufenacil vs. Glyphosate+saflufenacil	NA	0.0085*

\*, \*\*, \*\*\* , significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

NA: not applicable data recorded due to no glyphosate or saflufenacil in treatments.

Glyphosate residue accumulation in lentil seed is very important for lentil exporters because importing countries tend to reject lentil shipments if the glyphosate residue is over the MRL (Pratt, 2011). In the current study, average glyphosate residues did not exceed 2 ppm at the 30% application timing, nor did they exceed 4 ppm at 40% application timing (Figure 3.5). These values are not above the new EU MRL of 10 ppm, which was established in 2012. However, our results show that average glyphosate residue values do exceed the Canadian and Japanese limits of 4.0 and 2.0 ppm, respectively, and could exceed international CODEX levels of 5 ppm (Bryant Christie Inc., 2015). Therefore, it is imperative that growers do not apply glyphosate as a harvest aid when seed moisture content is above 40%. Our results also show that applications made prior to 40% seed moisture content consistently resulted in higher glyphosate residues. Glyphosate is a systemic herbicide that translocates slowly in the phloem and moves with nutrients to actively growing plant tissues (sucrose sinks) (Cessna et al., 1994, 2000; 2002; Duke and Powles, 2008). At early seed developmental stages, seeds are major sucrose sinks and glyphosate will translocate to those developing seeds. As the crop matures, the demand for sucrose from these sinks declines and less glyphosate is translocated to the developing seeds, resulting in reduced glyphosate residues.

An interesting finding was that there was no significant difference in glyphosate residues between the glyphosate alone treatment and the tank mix treatment of glyphosate+saflufenacil. Other research has also found that the addition of saflufenacil to glyphosate did not reduce seed residues, with an exception of 50% crop maturity in dry bean (McNaughton et al., 2015). Our results contrast with Ashigh and Hall (2010), however, who reported that glyphosate activity in plants was limited by adding saflufenacil, which can destroy plant phloem quickly. The

contrasting results may be due to lower sensitivity of pulse crops to saflufenacil compared to buckwheat, cabbage, and canola used in the Ashigh and Hall (2010) study.

Saflufenacil can be translocated in xylem and phloem (Ashigh and Hall, 2010; Soltani et al., 2010) and therefore, its residue is detectable in seeds. Currently, the lowest acceptable MRL for saflufenacil residue in lentil seed is 0.03 ppm as set by the European Union (Bryant Christie Inc., 2015). In this study, saflufenacil residues in lentil seeds generally increased with the earlier application timing of desiccants. Saflufenacil applied alone at 60% and 50% seed moisture resulted in unacceptable seed residue levels, exceeding 0.03 ppm in some cases. However, the current study found that saflufenacil residues were significantly lower in the tank mixture (with glyphosate) treatment, which did not exceed 0.03 ppm, regardless of application timing. This can be partially attributed to the lower rate of saflufenacil in the tank mixture (36 g a.i. ha<sup>-1</sup>) compared with saflufenacil applied alone (50 g a.i. ha<sup>-1</sup>). It is also possible that the reduction in the activity of saflufenacil within the plant might result from combining glyphosate with saflufenacil, which could adversely influence saflufenacil translocation. Ashigh and Hall (2010) reported that glyphosate limited the translocation of saflufenacil in glyphosate-susceptible canola. The authors suggested that glyphosate adversely impacts plant metabolism, resulting in reduced saflufenacil translocation. Nevertheless, McNaughton et al. (2015) reported that saflufenacil residues in dry bean did not change with the addition of glyphosate compared with the application of saflufenacil alone, which demonstrated that glyphosate did not have any effects on saflufenacil translocation in soybean. Based on these results, the interaction between saflufenacil and glyphosate in lentil needs to be further studied.

### **3.5 Conclusions**

As hypothesized, application of desiccants beyond 30% seed moisture content, when lentil was close to physiological maturity, did not influence seed yield or TSW. In addition, these application timings did not result in lentil seed samples exceeding residue levels of 2 ppm for glyphosate or 0.03 ppm for saflufenacil and thus, would not be problematic for seed exports. Although glyphosate residue levels were substantially lower in the tank mixture, adding saflufenacil to glyphosate did not reduce glyphosate residue in lentil seeds compared to glyphosate applied alone. It did, however, significantly reduce seed residues of saflufenacil.

Moreover, the tank mixture of glyphosate+saflufenacil exhibited improved crop desiccation compared with either glyphosate or saflufenacil applied alone. This tank mixture would also improve weed control over using either herbicide alone and offers two modes of action, which is important to delay the evolution of herbicide resistance. Given the results of this study, a tank mix of saflufenacil+glyphosate is recommended for crop desiccation and pre-harvest weed control in lentil over using either product alone. Regardless of the product chosen as a desiccant, our results show it is imperative to ensure applications of glyphosate or saflufenacil are not made prior to the 30% seed moisture stage.

## **4.0 Evaluation of Various Herbicides as Potential Desiccants in Lentil**

### **4.1 Introduction**

Canada is a major lentil producer, accounting for 39% of global lentil production. In the past decade, lentil production in Canada has increased from 1.1 to 2.0 million tonnes (FAOSTAT, 2015). Nearly all of Canada's lentils are produced in Western Canada, with 99% of the production occurring in Saskatchewan (Pulse Canada, 2014). Most of the increased lentil production is due to an increased number of hectares on which lentil is grown, owing largely to increased production efficiency (FAOSTAT, 2015; Statistics Canada, 2015). Yet despite the increased efficiency, harvesting lentil crops can still challenge growers.

Uniform seed maturity at harvest time is critical to lentil harvesting, and lentil are considered mature when the bottom third of the pods have changed color from yellow to brown (Saskatchewan Pulse Growers, 2011). This stage is considered the appropriate time to swath, desiccate, or apply pre-harvest herbicides to lentil crops. However, variations within a field can cause lentil plants to mature at different times. Moreover, lentil is an indeterminate plant with maturation occurring sequentially from lower pods to upper parts of the plant and thus, various stages of pod maturation can occur on the same plant (Saskatchewan Pulse Growers, 2011). These issues collectively produce patchy maturity at harvest, which can interfere with the harvesting operation and delay the crop harvest, resulting in poor harvesting efficiency, low seed yield and poor seed quality (Saskatchewan Pulse Growers, 2011).

To help reduce this variation, growers often use herbicides as harvest aids to desiccate the crop and ensure rapid and even dry-down of the crop seeds and foliage. The chemistry of desiccants and their application timing are critical because inappropriate application timing or rates can result in reductions in crop yield and quality (Bennett & Shaw, 2000; Boudreaux & Griffin, 2011), and can also leave unacceptable herbicide residue levels in seeds (Cessna et al., 1994; Wigfield et al., 1994; Cessna et al., 2000; Cessna et al., 2002). In western Canada, few herbicides have been registered as desiccants in lentil, and those that have include diquat, glyphosate, saflufenacil, and glufosinate (Saskatchewan Ministry of Agriculture, 2014). Glyphosate is the most popular desiccant in pulse production because it provides excellent control of late-emerging annual and perennial weeds, and it can improve crop desiccation (Soltani, 2013; McNaughton et

al., 2015). Glyphosate is a systemic herbicide that slowly kills plants by inhibiting 5-enolpyruvylshikimate-3-phosphate synthase (EPSPS), an enzyme critical for the production of aromatic amino acids (Devine et al., 1993; Cobb and Reade, 2010). Since glyphosate is translocated via phloem, it can move throughout the plant and tends to accumulate in seeds if glyphosate is applied at later crop growth stages (Cessna et al., 1994; Wigfield et al., 1994; Cessna et al., 2000; Cessna et al., 2002). However, the presence of glyphosate in lentil seed can be problematic, and concerns about glyphosate residues in lentil seed have caused trade restrictions in the past (Pratt, 2011). For example, Canadian lentils were rejected in 2011 by the European Union due to glyphosate residues exceeding 0.1ppm (Pratt, 2011), thereby limiting desiccant options for lentil growers.

Diquat is a contact herbicide that has traditionally been used as a desiccant in lentil crops. It can rapidly destroy plant tissues that it contacts, and has little-to-no translocation in the plant (Cobb and Reade, 2010; Saskatchewan Ministry of Agriculture, 2014). In Canada, glufosinate and saflufenacil are newly registered desiccants in lentil (Anonymous, 2014). Both herbicides are capable of translocation in the plant but similar to diquat, their movement is limited by rapid activity (Soltani et al., 2010). Apart from these registered herbicides, other potential herbicides may act as desiccants in lentil crops. Pyraflufen-ethyl is labeled as a contact desiccant in cotton and potatoes (Ivany, 2005; Nichino Europe Co. Limited, 2012), while flumioxazin provides rapid desiccation of dry bean (Soltani et al., 2013; Saskatchewan Ministry of Agriculture, 2013). Although there is no registration for pyraflufen-ethyl and flumioxazin application in lentil, they may have the potential as desiccants in lentil production.

There is currently limited information available on the effects of diquat, glufosinate ammonium, flumioxazin, saflufenacil, and pyraflufen-ethyl applied alone or in combination with glyphosate as desiccants for lentil dry-down. The addition of these contact herbicides to glyphosate could provide uniform crop desiccation and potentially improve weed control compared to if the herbicides are applied alone. Additionally, glyphosate residue may be reduced by the addition of these contact herbicides to glyphosate. Research is needed to identify herbicides or herbicide tank-mixes that leave minimal residues in the seed, provide rapid and uniform lentil crop desiccation, and have no effect on seed yield and quality. Thus, the objective of this study was to evaluate selected contact herbicides applied alone or in combination with

glyphosate for their ability to provide adequate lentil crop desiccation with minimal effects on seed yield, quality, and residues.

## **4.2 Hypotheses**

Harvest aids can provide adequate crop desiccation without impacting yield and seed quality. Second, increasing rate of harvest aids can improve desiccation performance without adverse effects on crop. In addition, glyphosate residues will be reduced when contact harvest aids are added to glyphosate.

## **4.3 Materials and Methods**

### **4.3.1 Experimental site and design**

Field experiments were conducted between 2012 and 2014 at Saskatoon and Scott, SK, Canada. Soil texture at Saskatoon ranged from clay to sandy loams, whereas the soil texture at Scott was a silty loam. The pH and organic matter content ranged from 7.5 to 7.9 and 2.4% to 4.5%, respectively, at Saskatoon. The Scott site had a pH of 5.3 to 6.8 and an organic matter content of 2.4% to 2.6%. The experimental design was a randomized complete block design with four replicates. Each block consisted of 21 herbicide treatments plus an unsprayed control. The herbicide treatments included pyraflufen-ethyl (10 g a.i. ha<sup>-1</sup> and 20 g a.i. ha<sup>-1</sup>), glufosinate (300 g a.i. ha<sup>-1</sup> and 600 g a.i. ha<sup>-1</sup>), flumioxazin (105 g a.i. ha<sup>-1</sup> and 210 g a.i. ha<sup>-1</sup>), saflufenacil (36 g a.i. ha<sup>-1</sup> and 50 g a.i. ha<sup>-1</sup>), and diquat (208 g a.i. ha<sup>-1</sup> and 415 g a.i. ha<sup>-1</sup>) each applied alone or in combination with glyphosate (900 g a.e. ha<sup>-1</sup>). Individual plot sizes were 2 m wide by 6 m long at Saskatoon, and 2 m wide by 5 m long at Scott.

### **4.3.2 Experimental procedure**

In the fall prior to plot establishment, the entire experimental area received an application of either ethalfluralin (Saskatoon, 1400 g a.i. ha<sup>-1</sup>) or imazethapyr (Scott, 13 g a.i. ha<sup>-1</sup>). A glyphosate burnoff (900 g a.e. ha<sup>-1</sup>) was made at both sites each spring before or immediately after seeding. Prior to seeding, a seed treatment of Apron Maxx RTA (0.73% fludioxonil; 1.10%

metalaxyl-M and S-isomer) was applied at a rate of 325 ml per 100 kg of lentil seed. Liquid Nodulator<sup>®</sup> inoculant (*Rhizobium leguminosarum* biovar *viceae*) was applied to seed at a rate of 2.76 ml kg<sup>-1</sup> in 2012, whereas Tag Team<sup>®</sup> Granular (*Rhizobium leguminosarum* and *Penicillium bilaii*) was applied at a rate of 2.8 kg ha<sup>-1</sup> in 2013 and 2014. Following the application of seed treatments, lentil was seeded into fallowed plots with a small plot drill equipped with single shoot hoe openers on 22 cm row spacing. Seeding depth was 3 cm, with a target plant density of 130 plants m<sup>-2</sup>. Plots were rolled at both sites immediately following lentil planting to provide a smooth and level surface for harvest. The cultivar CDC Maxim was used at all sites, as it is the most widely grown small red lentil cultivar in Western Canada.

Maintenance applications of herbicides were made at each site for post-emergence weed control. At Saskatoon, a tank mixture of imazamox plus imazethapyr (30 g a.i. ha<sup>-1</sup>) was applied between the 5<sup>th</sup> and 6<sup>th</sup> node stage of lentil development. At Scott, an in-crop application of quizalofop-p-ethyl (420 g a.i. ha<sup>-1</sup>) was made when lentil was at the 4<sup>th</sup> node stage. Any weeds not controlled by the herbicides were removed by hand to maintain weed-free plots. To prevent disease, prothioconazole (166 g a.i. ha<sup>-1</sup>) was applied at Saskatoon and boscalid (294 g a.i. ha<sup>-1</sup>) at Scott when lentil reached the early flowering stage.

The rates of herbicides used in the study are shown in Table 4.1. All herbicides were applied with the recommended adjuvant, either Merge<sup>®</sup> (50% surfactant; 50% petroleum hydrocarbons solvent) or Agral 90<sup>®</sup> (90% nonylphenoxy polyethoxy ethanol) (Table 4.1). Application timings, application dates, and environmental conditions are provided in Table 4.2. Herbicide treatments were applied with an air-pressurized tractor mounted sprayer equipped with shielding (110-015 AirMix nozzles, 275 kpa, 45 cm spacing) at Saskatoon, and with a CO<sub>2</sub>-pressurized bicycle sprayer (110-003 AirMix nozzles, 276 kpa, 25cm) at Scott. Both sprayers were calibrated to deliver 200 L ha<sup>-1</sup> of spray solution. All desiccant treatments were made when the crop was at 30% seed moisture content, with seed moisture content determined from randomly selected plants in border plots.

**Table 4.1** Herbicide treatments and rates for each herbicide treatment evaluated at Saskatoon and Scott, SK from 2012 to 2014.

Herbicide	Rate
	(g a.e. ha <sup>-1</sup> /g a.i. ha <sup>-1</sup> )
Control	0
Glyphosate	900
Pyraflufen-ethyl‡	10
Pyraflufen-ethyl+glyphosate‡	10 + 900
Pyraflufen-ethyl‡	20
Pyraflufen-ethyl+glyphosate‡	20 + 900
Glufosinate	300
Glufosinate+glyphosate	300 + 900
Glufosinate	600
Glufosinate+glyphosate	600+ 900
Flumioxazin¶	105
Flumioxazin+glyphosate¶	105 + 900
Flumioxazin¶	210
Flumioxazin+glyphosate¶	210 + 900
Saflufenacil§	36
Saflufenacil+glyphosate †	36 +900
Saflufenacil§	50
Saflufenacil+glyphosate†	50 +900
Diquat¶¶	208
Diquat+glyphosate¶¶	208 + 900
Diquat¶¶	415
Diquat+glyphosate¶¶	415 + 900

‡ Merge® at 1% v/v was added in pyraflufen-ethyl and the tank mixture of pyraflufen-ethyl+glyphosate treatment.

¶ Agral 90® at 0.25% v/v was added in flumioxazin treatment and the tank mixture of flumioxazin+glyphosate treatment.

§ Merge® at 1 L ha<sup>-1</sup> was added in saflufenacil treatment.

† Merge® at 0.5 L ha<sup>-1</sup> was added in the tank mixture of saflufenacil+glyphosate treatment.

¶¶ Agral 90® at 0.1% v/v was added in diquat and the tank mixture of diquat+glyphosate treatment.



**Table 4.2** Dates of application timings and environmental conditions (temperature, relative humidity and wind) for each treatment at Saskatoon and Scott, SK from 2012 to 2014.

Site	Year	Application timing	Application date	Temperature (°C)	Relative humidity %
Saskatoon	2012	30%	August 28	26.0	42.7
	2013	30%	August 19	30.1	30.5
	2014	30%	August 29	23.0	38.0
Scott	2012	30%	August 23	20.2	NA
	2013	30%	September 4	16.3	62.9
	2014	30%	August 22	13.8	46.9

NA: no applicable data was recorded

### 4.3.3 Data collection

Lentil plant density was determined in each plot two weeks after emergence by counting the number of plants in two randomly selected, one-meter rows. Visual ratings of desiccation progress were made at 3, 7, 14, and 21 days after application (DAA) based on the Canadian Weed Science Society 0 to 100 rating scale. On this scale, 80% represents commercially acceptable weed control, whereas 70 to 80% represents commercially acceptable weed suppression (Vanhala et al., 2004). The visual ratings at 3, 7, 14 and 21 DAA were used to calculate an area under the desiccant progress curve (AUDPC):

$$AUDPC = \left( \frac{D_1 + D_2}{2} \right) (t_2 - t_1) + \left( \frac{D_2 + D_3}{2} \right) (t_3 - t_2) + \left( \frac{D_3 + D_4}{2} \right) (t_4 - t_3) \quad [4.1]$$

where  $D_1$ ,  $D_2$ ,  $D_3$ , and  $D_4$  represent observed desiccation ratings at each evaluation day;  $t_1$ ,  $t_2$ ,  $t_3$ , and  $t_4$  represent the number of the days after each herbicide application (Jeger and Viljanen-Rollinson, 2001; Simko and Piepho, 2012). The four desiccation ratings were converted into a single relative value for reporting via the AUDPC equation, which models the progression of desiccation between ratings (McNaughton et al., 2015).

Lentils were harvested with a small plot combine when mature. Harvested seeds were cleaned using a dockage tester and weighed to determine clean seed yield. Final yield was determined by calculating clean yield and then adjusting to a moisture content of 13%. The weight of 1000 seeds (TSW) was determined by weighing 250 seeds and multiplying by four. Harvest straw moisture content was measured immediately after threshing each plot (except Scott in 2012) by

determining the fresh weight of plant straw, oven-drying the samples for 24 hours at 80°C, and then weighing the dried samples.

Glyphosate residue in seeds was measured at the Saskatoon and Scott locations in 2012 and 2013. Seed samples (250 g) from the unsprayed control and the glyphosate treatments were collected at 7 DAA. The samples were cleaned, placed into plastic bags and frozen at -20°C. Residue analyses (glyphosate and AMPA) were conducted by ALS Laboratories in Edmonton, AB, Canada, as described in Chapter 3.

#### **4.3.4 Statistical analysis**

PROC UNIVARIATE and Levene's test were used to examine normality and homogeneity of variance of the residuals, respectively (SAS Inst., 2014). Data were analyzed using the MIXED Procedure in SAS 9.3 (SAS Inst., 2014), with heterogeneous variance structures modeled within site-years as necessary. 'Repeated/group=options' was used to model heterogeneous variance for yield data because these data did not meet the assumptions of ANOVA after several transformations. In the mixed model, herbicide treatment was considered a fixed effect, while replication and its interaction with herbicide treatment were considered random effects. To determine whether data could be combined across site-years for analysis, the COVTEST option of PROC MIXED was used, with site-year as a random term in the model (SAS Inst., 2014). Where data could not be combined, data were analyzed within site-years, with site-year treated as a fixed effect. Means were separated using Tukey's HSD, with treatment differences declared significant at  $P \leq 0.05$ . Letter groupings were used to separate treatments and were created using the PDMIX800 macro in SAS (Saxton, 1998). Specific comparisons of interest were made between various herbicide treatments using single degree of freedom contrasts.

### **4.4 Results and Discussion**

#### **4.4.1 Lentil desiccation**

The interaction between site-year and herbicide treatment was significant and thus, data were analyzed within site-years (Table 4.3). At Saskatoon, most herbicide treatments tended to exhibit

better desiccation than the untreated control (Table 4.4). For example, glufosinate (300 or 600 g a.i. ha<sup>-1</sup>) or diquat (415 g a.i. ha<sup>-1</sup>) applied alone or in a tank mix with glyphosate resulted in desiccation progressing to the greatest extent, with some of these treatments showing 2- to 6-fold greater AUDPC than the untreated control. Treatments containing saflufenacil (36 or 50 g a.i. ha<sup>-1</sup>) or the lower rate of diquat (208 g a.i. ha<sup>-1</sup>) showed increased crop desiccation, as much as 4-fold greater than the untreated control (Table 4.4). Similar results were found for the tank mixture of pyraflufen-ethyl+glyphosate (3-fold increase) and flumioxazin+glyphosate (3-fold increase) (Table 4.4). Across all three years at the Saskatoon site, crop desiccation was least enhanced by glyphosate, pyraflufen-ethyl, or flumioxazin applied alone (Table 4.4). Contrasts showed that adding other contact herbicides to glyphosate significantly improved desiccation over glyphosate alone in all years, as did using higher rates of these herbicides (Table 4.4). In two of three years (2012 and 2014), adding glyphosate to the herbicide tank mixes improved desiccation relative to the contact herbicides alone. Based on the nature of glyphosate and the tank mix partners, these results are not unexpected; glyphosate is a slower acting desiccant than all other herbicides included in this study.

**Table 4.3** P-values derived from analysis of variance demonstrating area under desiccation progress curve (AUDPC), seed yield, thousand seed weight (TSW), straw moisture, and glyphosate residue (GR), as influenced by herbicide treatments at Saskatoon and Scott, SK from 2012 to 2014.

Source	AUDPC	Yield	TSW	Straw Moisture	GR
			P value		
Site-year (SY)	0.0621	0.0753	0.0699	0.0811	0.1203
Herbicide (H)	<.0001***	0.2547	0.4318	<.0001***	0.0044**
SY x H	<.0001***	0.3831	0.3516	0.0029**	0.0037**

\*, \*\*, \*\*\* , significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

**Table 4.4** Tukey's HSD means comparison of lentil areas under desiccation progress curve (AUDPC) at Saskatoon and Scott, SK from 2012 to 2014. Estimate statements represent differences between herbicide treatments in lentil desiccation.

Herbicide	Rate	AUDPC††					
		Saskatoon 2012	Saskatoon 2013	Saskatoon 2014	Scott 2012	Scott 2013	Scott 2014
Untreated	(g a.i./a.e. ha <sup>-1</sup> ) 0	218 G	999 F	691 L	920 E	1441 C	1143 BC
Glyphosate	900	700 D-F	1186 EF	836 J-L	1148 A-E	1596 A-C	1329 A-C
Pyraflufen-ethyl‡	10	538 F	1221 EF	788 KL	962 DE	1484 BC	1290 A-C
Pyraflufen-ethyl+ glyphosate‡	10+900	871 C-E	1358 C-E	942 G-K	1276 A-E	1527 A-C	1273 A-C
Pyraflufen-ethyl‡	20	563 F	1336 DE	856 I-L	999 C-E	1496 A-C	1232 A-C
Pyraflufen-ethyl+glyphosate‡	20+900	965 B-D	1361 C-E	1065 E-I	1149 A-E	1549 A-C	1235 A-C
Glufosinate	300	1531 A	1512 A-D	1258 B-E	1444 A-C	1668 AB	1555 A
Glufosinate+glyphosate	300+900	1532 A	1606 A-D	1324 A-D	1362 A-E	1610 A-C	1538 AB
Glufosinate	600	1614 A	1598 A-D	1518 A	1389 A-D	1694 A	1560 A
Glufosinate+glyphosate	600+900	1563 A	1620 A-C	1441 AB	1439 A-C	1670 AB	1537 AB
Flumioxazin¶	105	580 F	1205 EF	956 G-K	957 DE	1476 BC	1255 A-C
Flumioxazin+glyphosate¶	105+900	932 CD	1350 DE	964 G-K	1136 B-E	1522 A-C	1453 A-C
Flumioxazin¶	210	620 EF	1348 DE	909 H-K	1105 B-E	1492 A-C	1304 A-C
Flumioxazin+glyphosate¶	210+900	933 CD	1436 A-E	958 G-K	1185 A-E	1540 A-C	1130 C
Saflufenacil§	36	956 B-D	1366 BE	1011 F-J	1155 A-E	1435 C	1187 A-C
Saflufenacil+glyphosate†	36+900	1121 BC	1384 BE	1099 E-H	1335 A-E	1566 A-C	1343 A-C
Saflufenacil§	50	981 BC	1502 A-D	1103 E-H	1084 C-E	1536 A-C	1276 A-C
Saflufenacil+glyphosate†	50+900	1032 BC	1389 A-E	1109 D-H	1316 A-E	1546 A-C	1295 A-C
Diquat¶¶	208	1229 B	1515 A-D	1205 C-F	1370 A-D	1409 C	1176 A-C
Diquat+glyphosate¶¶	208+900	1091 BC	1499 A-D	1136 D-G	1400 A-D	1557 A-C	1300 A-C
Diquat¶¶	415	1535 A	1654 A	1413 A-C	1591 A	1526 A-C	1481 A-C
Diqua+glyphosate ¶¶	415+900	1527 A	1633 AB	1433 AB	1533 AB	1606 A-C	1425 A-C
HSD		274	270	216	451	208	397
Estimates							
Glyphosate vs. TM <sup>a</sup> +glyphosate		-457***	-278***	-311***	-165**	26	-24
TM <sup>a</sup> vs. TM <sup>a</sup> +glyphosate		-142***	-38	-45*	-107**	-47**	-21
TM <sup>a</sup> (low rate) vs. TM <sup>a</sup> (high rate)		-95***	-86***	-112***	-4	-40*	-10

\*, \*\*, \*\*\* , significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

†† Means within a column followed by the same letter are not significantly different on the basis of HSD<sub>0.05</sub>.

<sup>a</sup> TM denotes tank mix partners.

HSD denotes honest significant difference.

‡ Merge® at 1% v/v was added in pyraflufen-ethyl and the tank mixture of pyraflufen-ethyl+glyphosate treatment.

¶ Agral 90® at 0.25% v/v was added in flumioxazin treatment and the tank mixture of flumioxazin+glyphosate treatment.

§ Merge® at 1 L ha<sup>-1</sup> was added in saflufenacil treatment.

† Merge® at 0.5 L ha<sup>-1</sup> was added in the tank mixture of glyphosate+saflufenacil treatment.

¶¶ Agral 90® at 0.1% v/v was added in diquat and the tank mixture of diquat+glyphosate treatment.

Similar results were observed at the Scott site where in 2012, treatments containing diquat or glufosinate had the greatest desiccation efficiency, exhibiting a 57% greater AUDPC compared to the untreated control (Table 4.4). However, the other herbicide treatments did not significantly enhance desiccation. In 2013 and 2014, only the high rate of glufosinate (600 g a.i. ha<sup>-1</sup>) alone and in tank mixture with glyphosate provided significantly better desiccation (15% greater AUDPC) than the untreated control (Table 4.4). In two of three years (2012 and 2013), adding glyphosate to the herbicide tank mixtures improved desiccation compared with the contact herbicides alone. In contrast, adding contact herbicides to glyphosate only improved desiccation in one year (2012) compared with glyphosate applied alone. The rate of the contact herbicide at Scott had a relatively minor effect on desiccation.

The results of our study showed that adding contact herbicides to glyphosate facilitated lentil crop desiccation. In most years, there were benefits from tank-mixing glyphosate with other contact herbicides at both sites, and these tank mixes performed better than either the glyphosate or the tank mix partner applied alone. Soltani et al. (2013) also reported that the contact herbicides glufosinate, saflufenacil, diquat, carfentrazone-ethyl, and flumioxazin enhanced dry bean desiccation if tank mixed with glyphosate. As expected, the contact herbicides glufosinate and diquat produced rapid phytotoxic effects on plant tissues that came into direct contact with the active ingredient (Table 4.4), resulting in rapid and efficient desiccation of lentil plants. Our results are in agreement with Wilson and Smith (2002) and Soltani et al. (2013), both of whom reported increased dry bean desiccation with glufosinate applied at 80% of pod color change.

On the other hand, our results showed that glyphosate, pyraflufen-ethyl, and flumioxazin applied alone did not effectively enhance crop desiccation compared with the untreated control. The lack of effect for glyphosate is not surprising given that it requires translocation to actively growing metabolic sinks to inhibit plant growth and thus, exhibits slower crop dry-down than contact herbicides (Baylis, 2000; Duke and Powles, 2008; Schemenauer, 2011). Even though pyraflufen-ethyl and flumioxazin are labeled as contact herbicides (Valent Canada, Inc., 2009; Nichino Europe Co. Limited, 2012; Wisconsin Department of Natural Resources, 2012) and have been used as desiccants on potatoes and dry beans, they were not as effective as the other registered contact herbicides on lentil in this study. This might be explained by lower sensitivity of lentil to pyraflufen-ethyl and flumioxazin in comparison to glufosinate or diquat.

Nevertheless, our results suggest that these products will not provide effective desiccation of lentil crops.

#### **4.4.2 Seed yield and thousand seed weight**

Glyphosate applied alone or in combination with tank mix partners had no effect on seed yield or TSW (Table 4.3). Likewise, there was no significant interaction between herbicide treatment and site-years (Table 4.3), indicating that the absence of effects was consistent across all site-years (Table 4.5). Contrasts showed that adding glyphosate to other contact herbicides resulted in a statistically significant decrease in yield compared to when contact herbicides were applied alone (Table 4.5). However, the yield reduction was relatively minor at 6%. Besides, these tank mixture treatments did not reduce yield compared with the untreated control (Table 4.5). Seed yield and TSW were unaffected by the addition of contact herbicides to glyphosate compared to the glyphosate alone treatment. Likewise, higher rates of contact herbicides also did not affect lentil yield or TSW.

Our results suggest that glufosinate, saflufenacil, diquat, pyraflufen-ethyl, and flumioxazin applied alone or in combination with glyphosate will not affect lentil yield or TSW when applied at 30% seed moisture content. Similar results were found in other pulse crops when desiccants were applied close to, or at, crop maturity. For example, pre-harvest use of glyphosate, glufosinate, or paraquat had no adverse effects on seed yield and weight in dry bean (Wilson and Smith, 2002) and soybean (Ratnayake and Shaw, 1992; Ellis et al., 1998). In addition, Bennett and Shaw (2000a) reported that there was no difference in seed yield and TSW when glyphosate + sodium chlorate or paraquat + sodium chlorate were applied to soybean. Likewise, McNaughton et al. (2015) observed no significant reduction in dry bean yields when desiccants (glyphosate and saflufenacil) were applied at full maturity.

**Table 4.5** Tukey's HSD means comparison of seed yield and thousand seed weight (TSW) at Saskatoon and Scott, SK from 2012 to 2014. Estimate statements represent differences between herbicide treatments in seed yield and TSW.

Treatment	Rate (g a.i./a.e. ha <sup>-1</sup> )	Yield†† (kg ha <sup>-1</sup> )	TSW†† (g)
Untreated	0	3520.3	41.4
Glyphosate	900	3393.3	40.5
Pyraflufen-ethyl‡	10	3574.8	40.4
Pyraflufen-ethyl+glyphosate‡	10+900	3363.9	40.3
Pyraflufen-ethyl‡	20	3250.0	40.3
Pyraflufen-ethyl+glyphosate‡	20+900	3434.1	40.2
Glufosinate	300	3582.0	39.8
Glufosinate+glyphosate	300+900	3188.8	40.4
Glufosinate	600	3481.4	40.1
Glufosinate+glyphosate	600+900	3320.8	39.6
Flumioxazin¶	105	3361.7	40.6
Flumioxazin+glyphosate¶	105+900	3090.4	39.8
Flumioxazin¶	210	3336.0	40.8
Flumioxazin+glyphosate¶	210+900	3301.4	40.0
Saflufenacil§	36	3543.7	39.8
Saflufenacil+glyphosate†	36+900	3171.4	40.0
Saflufenacil§	50	3320.3	40.4
Saflufenacil+glyphosate†	50+900	3384.2	40.7
Diquat¶¶	208	3386.5	40.6
Diquat+glyphosate¶¶	208+900	3309.3	40.2
Diquat¶¶	415	3458.4	39.6
Diqua+glyphosate ¶¶	415+900	3346.8	40.5
HSD		NS	NS
<i>Estimates</i>			
Glyphosate vs. TM <sup>a</sup> +glyphosate		67.3	0.6
TM <sup>a</sup> vs. TM <sup>a</sup> +glyphosate		189.3***	0.2
TM <sup>a</sup> (low rate) vs. TM <sup>a</sup> (high rate)		-75.4	0.0

\*, \*\*, \*\*\* , significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

†† Means within a column followed by the same letter are not significantly different on the basis of HSD<sub>0.05</sub>.

<sup>a</sup>TM denotes tank mix partners.

HSD denotes honest significant difference.

NS denotes not significant at the 0.05 probability.

‡ Merge<sup>®</sup> at 1% v/v was added in pyraflufen-ethyl and the tank mixture of pyraflufen-ethyl+glyphosate treatment.

¶ Agral 90<sup>®</sup> at 0.25% v/v was added in flumioxazin treatment and the tank mixture of flumioxazin+glyphosate treatment.

§ Merge<sup>®</sup> at 1 L ha<sup>-1</sup> was added in saflufenacil treatment.

† Merge<sup>®</sup> at 0.5 L ha<sup>-1</sup> was added in the tank mixture of glyphosate+saflufenacil treatment.

¶¶ Agral 90<sup>®</sup> at 0.1% v/v was added in diquat and the tank mixture of diquat+glyphosate treatment.

In contrast, several other studies have reported reductions in soybean seed yield and quality with desiccants, such as paraquat and glyphosate (Whigham and Stoller, 1979; Azlin and McWhorter, 1981; Cerkauskas et al., 1982). Both Whigham and Stoller (1979) and Cerkauskas et al. (1982) noted reduced soybean yields when paraquat was applied before maturity. Azlin and McWhorter (1981) observed similar effects, reporting that yield and seed quality were reduced when glyphosate was used 3 to 4 weeks before harvest. The variability in the effects of desiccants on crop yield and quality can probably be attributed to when the herbicides were applied. The application of desiccants before physiological maturity may prevent photosynthesis for seed development or cause damage on immature seeds with herbicide residues (Retnayake and Shaw, 1992; Boudreaux and Griffin, 2011). The contact herbicides included in this study did not adversely affect lentil seed yield or weight and therefore, growers could apply these herbicides (if registered) at 30% seed moisture content without compromising lentil crop safety.

#### **4.4.3 Harvest straw moisture content**

The interaction between site-year and herbicide was significant (Table 4.3) and therefore, harvest straw moisture data were analyzed within site-years. At Saskatoon in 2013, glufosinate (300 or 600 g a.i. g ha<sup>-1</sup>) and diquat (415 g a.i. ha<sup>-1</sup>) alone or tank mixed with glyphosate resulted in a 27% reduction in straw moisture content compared with the untreated control (Table 4.6). At Saskatoon in 2014, glufosinate (300 or 600 g a.i. g ha<sup>-1</sup>) and diquat (415 g a.i. ha<sup>-1</sup>) alone or in mixture with glyphosate, as well as saflufenacil (36 or 50 g a.i. ha<sup>-1</sup>) or flumioxazin (105 g a.i. ha<sup>-1</sup>) tank mixed with glyphosate, effectively decreased straw moisture content by 17 to 35% compared to the untreated control (Table 4.6). Across both years at Saskatoon, the other herbicides generally did not differ from the untreated control (Table 4.6). Desiccants had no effect ( $P>0.05$ ) on straw moisture content at Saskatoon in 2012. With the exception of the glufosinate treatments, desiccants had no effect on straw moisture content compared with the untreated control at the Scott site. Plots that received glufosinate at Scott exhibited a 67 and 43% reduction in straw moisture content in 2013 and 2014, respectively (Table 4.6).



**Table 4.6** Tukey's HSD means comparison of harvest straw moisture at Saskatoon and Scott, SK from 2012 to 2014. Estimate statements represent differences between herbicide treatments in harvest straw moisture.

Herbicide	Rate (g a.i./a.e. ha <sup>-1</sup> )	Straw moisture				
		Saskatoon 2012	Saskatoon 2013	Saskatoon 2014	Scott 2013	Scott 2014
		(%)				
Untreated	0	54.9	47.6 A	55.5 A	33.4 A	62.5 A
Glyphosate	900	45.5	48.0 A	47.9 A-D	18.9 A-E	43.6 AB
Pyraflufen-ethyl‡	10	52.8	45.2 A-C	55.6 A	31.2 A-C	43.4 AB
Pyraflufen-ethyl+glyphosate‡	10+900	42.6	45.1 A-C	49.8 A-D	20.6 A-E	51.5 AB
Pyraflufen-ethyl‡	20	48.4	40.5 A-D	53.8 AB	26.5 A-D	49.2 AB
Pyraflufen-ethyl+glyphosate‡	20+900	46.6	44.7 A-C	47.1 A-E	21.0 A-E	54.6 AB
Glufosinate	300	41.0	41.1 A-D	42.4 D-G	11.7 C-E	43.3 AB
Glufosinate+glyphosate	300+900	42.8	33.4 CD	36.4 F-H	12.2 B-E	45.5 AB
Glufosinate	600	43.4	34.9 B-D	35.1 GH	9.9 DE	49.5 AB
Glufosinate+glyphosate	600+900	45.3	29.5 D	30.6 H	9.8 E	35.5 B
Flumioxazin¶	105	46.8	48.3 A	47.8 A-D	31.2 A-C	48.6 AB
Flumioxazin+glyphosate¶	105+900	48.5	43.1 A-C	45.8 B-E	18.5 A-E	46.1 AB
Flumioxazin¶	210	50.4	43.2 A-C	52.0 A-C	28.1 A-C	50.8 AB
Flumioxazin+glyphosate¶	210+900	46.3	39.8 A-D	47.4 A-E	25.9 A-E	53.8 AB
Saflufenacil§	36	50.5	43.1 A-C	49.3 A-D	32.6 AB	57.0 AB
Saflufenacil+glyphosate†	36+900	43.7	39.1 A-D	41.8 D-G	22.0 A-E	47.9 AB
Saflufenacil§	50	48.4	36.7 A-D	48.7 A-D	22.7 A-E	46.5 AB
Saflufenacil+glyphosate†	50+900	48.4	43.4 A-C	44.8 C-F	15.5 A-E	43.7 AB
Diquat¶¶	208	45.2	39.4 A-D	46.6 B-E	32.6 AB	48.3 AB
Diquat+glyphosate¶¶	208+900	48.2	46.5 AB	45.1 B-F	20.3 A-E	41.6 AB
Diquat¶¶	415	44.5	34.6 B-D	38.9 E-H	23.6 A-E	44.9 AB
Diquat+glyphosate ¶¶	415+900	43.7	34.2 CD	36.4 F-H	25.7 A-E	50.1 AB
HSD		NS	23.0	8.9	22.5	23.3
<i>Estimate</i>						
Glyphosate vs. TM <sup>a</sup> +glyphosate		0.2	8.2***	5.4**	-0.2	-3.4
TM <sup>a</sup> vs. TM <sup>a</sup> +glyphosate		1.5	0.8	4.5***	5.9**	1.1
TM <sup>a</sup> (low rate) vs. TM <sup>a</sup> (high rate)		-0.3	4.3***	2.6***	2.4	-0.5

\*, \*\*,\*\*\*, significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

†† Means within a column followed by the same letter are not significantly different on the basis of HSD<sub>0.05</sub>.

<sup>a</sup>TM denotes tank mix partners.

HSD denotes honest significant difference.

NS denotes not significant at the 0.05 probability.

‡ Merge<sup>®</sup> at 1% v/v was added in pyraflufen-ethyl and the tank mixture of pyraflufen-ethyl+glyphosate treatment.

¶ Agral 90<sup>®</sup> at 0.25% v/v was added in flumioxazin treatment and the tank mixture of flumioxazin+glyphosate treatment.

§ Merge<sup>®</sup> at 1 L ha<sup>-1</sup> was added in saflufenacil treatment.

† Merge<sup>®</sup> at 0.5 L ha<sup>-1</sup> was added in the tank mixture of glyphosate+saflufenacil treatment.

¶¶ Agral 90<sup>®</sup> at 0.1% v/v was added in diquat and the tank mixture of diquat+glyphosate treatment.

Contrasts produced similar results to AUDPC at both sites across the three years of the study. At Saskatoon in 2013 and 2014, the addition of contact herbicides to glyphosate decreased straw moisture by an average of 6.8%, while no difference was observed at Scott in either year (Table 4.6). Similarly, when glyphosate was added to the various contact herbicides, straw moisture content was significantly lower compared to when they were used alone in two site-years, although the trend was numerically consistent across all site-years (Table 4.6). Thus, the addition of glyphosate to the contact herbicides improved crop desiccation and reduced straw moisture content at harvest, thereby facilitating improved harvest efficiency. The low rates of each herbicide resulted in higher straw moisture content than the high rates in two of the five site-years (Table 4.6).

Generally, glufosinate and diquat had the greatest and most consistent effect on reducing straw moisture content, which corresponded well with the AUDPC (Table 4.4 and 4.6). The enhancement of straw desiccation by applying glufosinate and diquat was also observed in potato (Ivany and Sanderson, 2001) and alfalfa (Moyer et al., 1996). Both of these studies reported that diquat was more effective than glufosinate, but the advantage decreased at later crop growth stages (Moyer et al., 1996; Ivany and Sanderson, 2001). In contrast, the other herbicides in our study had inconsistent effects on straw dry-down. It is possible that lentil is less tolerant to glufosinate and diquat than the other contact herbicides.

Our results showed that pyraflufen-ethyl and flumioxazin had no effects on straw dry-down; thus, they may not be good options to improve harvest efficiency. It is also possible that spray coverage differed between the various herbicides included in this study. Good spray coverage of contact herbicides is required to achieve adequate crop desiccation due to their limited translocation (Saskatchewan Ministry of Agriculture, 2014). Effective straw desiccation with some herbicides may only be achieved by changing the water volume, nozzle type, boom height or ground speed to provide better spray coverage. More research is required to determine if this improves lentil desiccation with pyraflufen-ethyl and flumioxazin.

#### **4.4.4 Glyphosate residue**

Glyphosate residues varied between site-years and therefore, glyphosate residue data were analyzed separately within site-years. None of the herbicide treatments exceeded 4.0 ppm of

glyphosate (MRL set by Canada) at Saskatoon in 2012. In treatments where glyphosate was tank mixed with glufosinate or diquat, glyphosate residues were significantly lower than when glyphosate was applied alone, and did not exceed 2.0 ppm (MRL set by Japan) (Table 4.7). The addition of glufosinate (300 or 600 g a.i. ha<sup>-1</sup>), saflufenacil (36 g a.i. ha<sup>-1</sup>), or diquat (208 or 415 g a.i. ha<sup>-1</sup>) to glyphosate decreased glyphosate residues between 43% and 73% compared to glyphosate alone (Table 4.7). Likewise, at Scott in 2012, glufosinate (600 g a.i. ha<sup>-1</sup>) or diquat (208 or 415 g a.i. ha<sup>-1</sup>) tank mixed with glyphosate resulted in residue levels that did not exceed 4.0 ppm or 2.0 ppm, respectively. Not surprisingly, contrasts showed that glyphosate residues were significantly lower (1.2 ppm, on average) when contact herbicides were added to glyphosate at Saskatoon in 2012. A similar 1.0 ppm reduction was observed at Scott in 2012 when contact herbicides were added to glyphosate, but the reduction was not statistically significant.

In contrast, pyraflufen-ethyl (10 or 20 g a.i. ha<sup>-1</sup>), saflufenacil (50 g a.i. ha<sup>-1</sup>) and flumioxazin (105 or 210 g a.i. ha<sup>-1</sup>) did not affect glyphosate residue levels compared with glyphosate applied alone (Table 4.7). There were no differences in glyphosate residue between desiccant treatments at Saskatoon or Scott in 2013; none of the treatments resulted in unacceptable herbicide residues. In addition, glyphosate residue was unaffected by herbicide rate (Table 4.7). A lack of differences in glyphosate residue between treatments may result from reduced translocation of glyphosate to lentil seeds in 2013. Reduced translocation may be related to lower seed moisture contents at the time of application in 2013 (32% and 35% for Saskatoon and Scott, respectively) compared to 2012 (35% and 40% for Saskatoon and Scott, respectively). Decreased glyphosate residue with lower seed moisture at application was also observed in wheat (*Triticum aestivum* L.), field pea (*Pisum sativum* L.), barley (*Hordeum vulgare* L.), flax (*Linum usitatissimum* L.) and canola (*Brassica rapa* L.) (Cessna et al., 1994, 2000; 2002).

**Table 4.7** Tukey's HSD means comparison of glyphosate residue (GR) at Saskatoon and Scott, SK, in 2012 and 2013. Estimate statements represent differences between herbicide treatments in glyphosate residue.

Treatment	Rate	GR ††			
		Saskatoon 2012	Saskatoon 2013	Scott 2012	Scott 2013
	(g a.i./a.e. ha-1)	(ppm)			
Glyphosate	900	3.5 A	0.7	3.7 AB	0.2
Pyraflufen-ethyl + Glyphosate‡	10+900	3.1 AB	0.1	3.7 AB	0.1
Pyraflufen-ethyl + Glyphosate‡	20+900	2.5 A-D	1.1	2.6 A-C	0.1
Glufosinate + Glyphosate	300+900	1.6 DE	0.1	2.3 A-C	0.1
Glufosinate + Glyphosate	600+900	1.7 C-E	0.2	1.2 BC	0.1
Flumioxazin + Glyphosate¶	105+900	3.4 A	1.7	4.8 A	0.1
Flumioxazin + Glyphosate¶	210+900	3.8 A	0.3	4.4 A	0.1
Saflufenacil + Glyphosate§	36+900	2.0 B-D	0.8	3.6 AB	0.1
Saflufenacil + Glyphosate†	50+900	2.8 A-C	0.3	3.3 A-C	0.1
Diquat + Glyphosate¶¶	208+900	1.2 EF	0.3	1.1 BC	0.1
Diquat + Glyphosate¶¶	415+900	0.7 F	0.1	0.5 C	0.1
HSD		1.3	NS	2.9	NS
Estimates					
Glyphosate vs. TM <sup>a</sup> +glyphosate		1.2**	0.1	1	0.0
TM <sup>a</sup> +glyphosate (low rate) vs. TM <sup>a</sup> +glyphosate (high rate)		0.0	0.2	0.7	0.0

\*, \*\*,\*\*\*, significant at the 0.05, 0.01, and 0.001 probability levels, respectively.

†† Means within a column followed by the same letter are not significantly different on the basis of HSD0.05.

<sup>a</sup>TM denotes tank mix partners.

HSD denotes honest significant difference.

NS denotes not significant at the 0.05 probability.

‡ Merge® at 1% v/v was added in pyraflufen-ethyl and the tank mixture of pyraflufen-ethyl+glyphosate treatment.

¶ Agral 90® at 0.25% v/v was added in flumioxazin treatment and the tank mixture of flumioxazin+glyphosate treatment.

§ Merge® at 1 L ha-1 was added in saflufenacil treatment.

† Merge® at 0.5 L ha-1 was added in the tank mixture of glyphosate+saflufenacil treatment.

¶¶ Agral 90® at 0.1% v/v was added in diquat and the tank mixture of diquat+glyphosate treatment.

Glyphosate residue is an important consideration for exporters because unacceptable glyphosate residue levels can cause rejection of shipments by importers. Currently, the two lowest MRLs are 2 ppm and 4 ppm, set by Japan and Canada, respectively (Bryant Christie Inc., 2015). Results from this study suggest that using glyphosate as a desiccant can result in unacceptable glyphosate residue levels (Japan MRL), even if it is applied at the recommended 30% seed moisture content. However, tank mixing glufosinate (600 g a.i. ha<sup>-1</sup>) or diquat (208 or 415 g a.i. ha<sup>-1</sup>) with glyphosate consistently provided significant reductions in glyphosate residue such that residues typically did not exceed 2 ppm (Table 4.7). Other treatments failed to reduce glyphosate residues and some, such as flumioxazin, resulted in higher levels of glyphosate residues in lentil seed (Table 4.7). Based on this, producers are unable to limit glyphosate residues in lentil seed by tank mixing glyphosate with saflufenacil. Moreover, results presented in Chapter (2) also showed little reduction in glyphosate residues when it was tank mixed with saflufenacil, regardless of seed moisture content. Therefore, this tank-mix should not be used with the intention of reducing glyphosate residues. This contrasts with previous studies on other crops including in buckwheat, cabbage, and canola, which have shown that saflufenacil reduced the activity of glyphosate (Ashigh and Hall, 2010). Lentil may be inherently less sensitive to saflufenacil than the other crops, as shown by Soltani et al. (2010).

The contact herbicides used in this study were hypothesized to produce faster crop desiccation than glyphosate, thereby trapping glyphosate in the leaves of treated plants, slowing translocation and reducing glyphosate residue levels. Our results indicated this was only possible in tank mixtures with glufosinate and diquat, which resulted in the lowest glyphosate residues among all treatments containing glyphosate. This was probably a product of limited glyphosate movement in lentil due to the very quick herbicidal action of the contact herbicides (Wehtje et al., 2008; Bethke et al, 2013). The highest glyphosate residues observed in this study were found in the tank mix of flumioxazin and glyphosate and therefore, this mixture will not help limit glyphosate residues.

## **4.5 Conclusions**

The results of this study showed that glufosinate (600 g a.i. ha<sup>-1</sup>) and diquat (415 g a.i. g ha<sup>-1</sup>) applied alone or tank mixed with glyphosate consistently provided the greatest crop desiccation

without any adverse effects on lentil seed yield and weight. Perhaps more importantly, these treatments also had acceptable glyphosate residue levels, generally < 2 ppm. Saflufenacil (36 or 50 g a.i. ha<sup>-1</sup>) applied alone or in mixture with glyphosate often provided better desiccation compared to the untreated control, but residue levels were unacceptable (> 2 ppm) in some site-years. Pyraflufen-ethyl and flumioxazin, applied alone or in mixture with glyphosate, provided the slowest desiccation, and did not significantly reduce glyphosate residues compared to glyphosate applied alone. As hypothesized, tank mixing contact herbicides with glyphosate generally improved lentil desiccation without yield losses or reductions in seed weight. More specifically, the traditional desiccants glufosinate and diquat provided the greatest reduction in glyphosate residue, the fastest crop desiccation, and did not affect seed yield and weight. It is recommended that growers use one of these two contact herbicides for lentil desiccation, though consideration must be given to the efficacy of weed control provided by each mixture. However, further research is needed in that regard.

## 5.0 General Discussion

### 5.1 The use of desiccants in lentil

Results presented in this thesis demonstrate the importance of appropriate application timing of desiccants in facilitating crop desiccation. Proper timing of desiccants maintained crop yield, seed quality, and low levels of herbicide residues in seeds. Both lentil yield and thousand seed weight (TSW) were reduced by desiccants applied at the earliest crop growth stages (Chapter 2). However, yield and seed weight were not negatively influenced when desiccants were applied beyond 50% seed moisture content. Results from Chapter 3 also confirmed that yield and TSW were not compromised if desiccants were used at the correct maturity. These findings proved the first hypothesis that the application of desiccants at or close to crop maturity would not affect seed yield or TSW in lentil, supporting product recommendations. Several studies in soybean (*Glycine max* L.) (Whigham and Stroller, 1979; Cerkauskas et al., 1982; Ratnayake and Shaw, 1992; Ellis et al., 1998; Bennett and Shaw, 2000a; Boudreaux and Griffin, 2011), dry bean (*Phaseolus vulgaris* L.) (Wilson and Smith, 2002; McNaughton et al., 2015), and wheat (*Triticum aestivum* L.) (Darwent et al., 2000; Yenish and Yong, 2000) also showed no detrimental effects of using desiccants on crop yield and quality, unless the desiccants were applied before crop physiological maturity. These authors attributed yield loss and seed quality reductions to crop immaturity at the early applications, resulting in reduced plant growth and seed development (Whigham and Stroller, 1979; Cerkauskas et al., 1982; Ratnayake and Shaw, 1992; Ellis et al., 1998; Bennett and Shaw, 2000a; Darwent et al., 2000; Wilson and Smith, 2002; Boudreaux and Griffin, 2011).

Another hypothesis presented in this thesis was that herbicide residues would decrease with later application of desiccants, and this was confirmed by the results presented in Chapter 2. Glyphosate residues declined with delayed applications of desiccants (>40% seed moisture content), and were below 2ppm (the lowest MRL of glyphosate set by Japan) at 30% or lower seed moisture (Chapter 2). The declines in glyphosate residues that we observed at later growth stages suggest that proper application stage (close to crop maturity) will not leave unacceptable glyphosate residues in lentil seeds. In fact, the data presented in Chapter 3 also showed that glyphosate residues were acceptable (<4 ppm) even if glyphosate was applied in a tank mixture

with several other desiccants. All applications in that trial were made at 30% seed moisture content. Similar trends have also been reported in other crops, such as wheat (Cessna et al., 1994), field pea (*Pisum sativum* L.), barley (*Hordeum vulgare* L.), flax (*Linum usitatissimum* L.) (Cessna et al., 2002), canola (*Brassica rapa* L.) (Cessna et al., 2000), and dry bean (Soltani et al., 2013). Glyphosate can be translocated readily within plants and concentrate in areas with high metabolic activities. For minimum glyphosate residues in lentil seed, desiccant applications should be delayed to as close to crop maturity as possible, such as 30% or less seed moisture content. (Cessna et al., 1994, 2000; 2002).

We also observed a reduction in saflufenacil residues when it was applied at lower seed moisture contents (Chapter 2). Saflufenacil applied alone at 50% and 60% seed moisture led to saflufenacil residues exceeding the acceptable level (0.03 ppm) imposed by the European Union (Chapter 2). Similar findings were reported in dry bean (McNaughton et al., 2015). As with glyphosate, less saflufenacil likely was translocated to seeds due to the reduced demand for sucrose as the crop matures (McNaughton et al., 2015). Although there was no reduction in yield or seed weight below 40% seed moisture, herbicide residues were only reduced to below the acceptable level (EU) when applications were made below 30% seed moisture content (Chapter 2). Thus, early (>30% seed moisture) application of desiccants is risky and should be avoided by producers. The results support the product labels, all of which state that desiccants should be applied at 30% seed moisture content or less. Our data indicate there is very little flexibility to apply desiccants early in order to accelerate crop dry-down without effects on yield, seed weight, or herbicide residues.

Another important part of this thesis was to evaluate whether application of contact herbicides alone or tank-mixed with glyphosate could provide adequate crop dry-down and effectively reduce glyphosate residues in the seed. On the whole, results suggest that the addition of contact herbicides to glyphosate improved crop desiccation without yield loss or reduced thousand seed weight, but most treatments did not effectively decrease glyphosate residues relative to the glyphosate alone treatment (Chapter 2 and 3). Seed yield and weight were likely unaffected because crop development was terminated when desiccants were applied, resulting in minimal effects on seed yield and weight (Whigham and Stroller, 1979; Cerkauskas et al., 1982; Ratnayake and Shaw, 1992; Ellis et al., 1998; Bennett and Shaw, 2000a; Wilson and Smith, 2002; Boudreaux and Griffin, 2011). Compared with the untreated control, glufosinate and



diquat provided the fastest crop dry-down and reduced glyphosate residues without impacting seed yield and weight (Chapter 3). The reduced glyphosate residues in seeds may be due to the rapid action of these two contact herbicides (Wehtje et al., 2008; Bethke et al, 2013).

Saflufenacil also accelerated crop desiccation, but it did not have a positive impact on glyphosate residues and in some cases (2012), led to unacceptable glyphosate residues (Chapter 2 and 3).

Pyraflufen-ethyl and flumioxazin did not effectively desiccate the crop and these treatments, when combined with glyphosate, did not help lower glyphosate residues. It is possible that lentil plants might be less sensitive to saflufenacil, pyraflufen-ethyl and flumioxazin than the traditional desiccants (glufosinate and diquat). Soltani et al. (2005, 2011) and Ivany (2005) reported different sensitivity of crops to flumioxazin, saflufenacil, and pyraflufen-ethyl, respectively. Soltani et al. (2005) attributed the differences to seed size and differential gene pools of various market classes due to different origins, and demonstrated that the larger seeded dry beans were more tolerant to flumioxazin than the smaller seeded dry beans.

## **5.2 Management implications**

The results of these studies demonstrate that early application of desiccants prior to full crop maturity caused reductions in seed yield and quality. More importantly, herbicide residues at these application timings exceeded the lowest acceptable MRLs for glyphosate and saflufenacil. This is problematic for international trade, and could cause economic losses if importing countries reject exports. Thus, lentil producers must carefully follow product labels to decrease the risks associated with early application of desiccants. Although this can be challenging, identifying the proper stage for desiccant application could be achieved by calculating seed moisture content, as was done in this thesis. Growers often prefer using visual indicators of plant maturity because this is quicker and more efficient. For example, applications should be made when 15% of the pods are changing from yellow to brown, coinciding with roughly 30% seed moisture content. However, these visual indicators are very subjective, and do not always provide accurate assessments of seed moisture content, which can lead to early applications. In such cases, it is recommended that growers obtain the moisture content of the seed prior to the application of desiccants. It may also be necessary, and perhaps even critical, to recruit experienced agronomists to help growers determine the appropriate timing for the application of

desiccants. It is also recommended that producers familiarize themselves with the MRLs of importing countries to avoid trade issues.

The results of this study indicated that tank mixing contact herbicides with glyphosate was beneficial to facilitate crop desiccation without adverse effects on seed yield and quality. Glufosinate and diquat alone or in mixture with glyphosate had the most consistent desiccation and acceptable glyphosate residues. In addition, glyphosate is an excellent pre-harvest herbicide to control late-emerging weeds, but glufosinate and diquat usually cannot provide enough weed control (Schemenauer, 2011). Therefore, using tank mixtures (glufosinate+glyphosate or diquat+glyphosate) would be a better option for growers with regard to the presence of late emerging weeds in field. The application of glyphosate alone may not provide rapid crop desiccation. Alternatively, tank mixing contact herbicides (only glufosinate and diquat) with glyphosate can reduce glyphosate residue at 30% seed moisture (Table 4.7). Although other contact herbicide did not significantly decrease glyphosate residue, glyphosate residue surpassed only the MRL of Japan and thus, we cannot recommend this practice to growers if they export lentils to Japan. It is possible that these treatments would have reduced glyphosate residues if applied earlier (>30% seed moisture), but this was not evaluated in this thesis and future research should be conducted in that regard. In addition, the treatments including pyraflufen-ethyl and flumioxazin did not show significant advantages for lentil crop desiccation, nor did they reduce glyphosate residue. These findings suggest that these three herbicides are not good desiccant options for growers.

### **5.3 Future research**

There was only one lentil cultivar included in this study and it is possible that lentil cultivars may respond differently to desiccants. More research is needed to evaluate other lentil cultivars to confirm if there is a consistent effect of desiccants for lentil dry-down. In addition, the two trials in this thesis were conducted under weed-free field conditions, but the response of the crop to desiccants may change under weedy conditions. This is particularly true in the case of contact herbicides, which may be greatly impacted by dense weed stands due to poor spray coverage. Additional research is needed to evaluate desiccants under both weedy and weedy-free fields to identify the stability of yield and seed quality, and glyphosate residue levels. Further research

should also be conducted to determine the impact on weed control of the desiccants included in this thesis. It is likely that many of the contact herbicides would exhibit poor weed control, especially for perennial weeds.

In addition, both study locations (Saskatoon and Scott) are in Saskatchewan, and the results of current study showed crop desiccation progression varied among site-years due to different environmental conditions. Other studies have reported that environmental conditions significantly influenced crop responses to desiccants due to soil texture, temperature and rainfall (Moyer et al., 1996; Willson and Smith, 2002). More research in other areas of Canada should be included in future studies as only two sites could be included in this due to logistical constraints. Future studies with several site-years of data would provide more accurate information on the efficiency of desiccants.

Since glyphosate residue is a main concern for Canadian exporters, future research should be conducted to evaluate if a lower rate of glyphosate ( $450 \text{ g a.i. ha}^{-1}$ ) in mixture with contact herbicides can provide both adequate crop desiccation and acceptable glyphosate residues in seeds. Lower glyphosate residues in wheat, field pea, barley, flax, and canola were observed as the dosage of glyphosate decreased (Cessna et al., 1994, 2000; 2002). It is possible that higher rates of contact herbicides applied with a lower rate of glyphosate may provide adequate crop dry-down and weed control.

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